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Characterization of Hanford High Level Waste Glass Round Robin Samples

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EXECUTIVE SUMMARY

This report presents the results of glass characterization performed at the Savannah River National Laboratory (SRNL) in support of a U.S. Department of Energy – Office of River Protection round robin study of simulated high-level waste glasses produced in small scale melters. The round robin study is coordinated by the Pacific Northwest National Laboratory (PNNL). SRNL received a series of glass samples from PNNL as part of this study, including CJS-2018-2, HLW-HCr-16-X(IV) [also labeled CJS-2018_1], HLW-HCr-16-Y(VI) [also labeled CJS-2018_2], HLW-HCr-16-X (II), and HLW-HCr-16-Y (I).

SRNL performed chemical composition measurements, heat treatments, crystal fraction measurements via X-ray diffraction, viscosity determinations, and chemical durability evaluations using the Product Consistency Test. The results of this work are detailed in this report. PNNL will use these data for comparisons with the results provided by other laboratories participating in the round robin study.

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LIST OF ABBREVIATIONS

AD	Acid Dissolution
ar	as-received
ARM-1	Approved Reference Material
BCH	waste compliance plan Batch 1 glass
BDL	Below Detection Limit
CCC	Canister Centerline Cooled
DOE	U.S. Department of Energy
EA	Environmental Assessment
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
HLW	High Level Waste
KH	potassium hydroxide fusion
LM	Lithium Metaborate fusion
LRM	Low-level Reference Material
NIST	National Institute of Standards and Technology
ORP	Office of River Protection
PCT	Product Consistency Test
PF	sodium Peroxide Fusion
PNNL	Pacific Northwest National Laboratory
PSAL	Process Science Analytical Laboratory
SRNL	Savannah River National Laboratory
wt %	weight percent
WTP	Waste Treatment and Immobilization Plant
XRD	X-ray Diffraction

1.0 Introduction

The U.S. Department of Energy – Office of River Protection (DOE-ORP) is administering a high-level waste (HLW) round robin glass property evaluation among three laboratories: Pacific Northwest National Laboratory (PNNL), the Catholic University of America Vitreous State Laboratory, and Savannah River National Laboratory (SRNL). The objectives of this round robin study are: 1) to determine whether the properties of glasses produced in melter tests performed at PNNL and VSL match within appropriate uncertainties, and 2) to determine whether the results of simulated Hanford HLW glass properties measured at PNNL, VSL, and SRNL match within appropriate uncertainties.

PNNL and VSL each performed a melter run using an agreed upon feed, producing glass of the targeted composition for the round robin study. SRNL previously reported the results of characterization samples of the glasses produced from these melter runs.[1] PNNL has requested that SRNL perform additional analyses of glass samples as part of continuing efforts for this round robin evaluation.

Two shipments of glass samples were received from PNNL. The samples were characterized at SRNL and are described in this report. In addition, further characterization of glass samples received in 2014 was completed at the request of PNNL and is described herein.

2.0 Experimental Procedure

2.1 Quality Assurance

The work performed at SRNL is controlled by a Task Technical and Quality Assurance Plan.[2] Data for these analyses is recorded in SRNL's electronic laboratory notebook system, in experiment C3489-00079-27. Requirements for performing reviews of technical reports and the extent of review are established in Savannah River Site Manual E7, Procedure 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

2.2 Glass Samples for Characterization

The first shipment from PNNL contained a glass sample designated CJS-2018-2, and was received in November 2018. The second shipment contained glass samples labeled CJS-2018_1 and CJS-2018_2, and was received in June 2019. PNNL gave secondary labels for these samples on the chain of custody form: HLW-HCr-16-X(IV) for CJS-2018_1 and HLW-HCr-16-Y(VI) for CJS-2018_2. The secondary labels were used at SRNL to avoid confusion with the similar label of the sample received in November 2018. A suite of characterization techniques was requested for the first sample shipment. Only a subset of the characterization techniques was requested for the second shipment. Details of the characterization methods used are described, chronologically, in the following sections.

During the course of this work, PNNL requested that SRNL measure the viscosity of earlier round robin samples[1] received in June 2014. These samples are labeled HLW-HCr-16-X (II) and HLW-HCr-16-Y (I).

2.3 November 2018 Sample Characterization

Approximately 180 g of a glass identified as CJS-2018-2 were received from PNNL. The glass was received in the as-fabricated state; that is, no additional heat treatment has been performed after production in the melter.

2.3.1 Chemical Composition Measurements

The chemical composition of the as-received glass sample was measured at SRNL's Process Science Analytical Laboratory (PSAL) following an experimental plan.[3] Quadruplicate samples of the glass were submitted to PSAL. Each of the four samples was prepared in duplicate by each of four preparation methods, including acid dissolution (AD),[a] lithium metaborate fusion (LM),[4] sodium peroxide fusion (PF),[5] and potassium hydroxide fusion (KH).[6] Samples dissolved by the first three preparation methods (AD, LM, and PF) were measured using Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES). Samples dissolved by KH were measured using Ion Chromatography (IC). Each dissolved sample was measured twice, with the instrument being calibrated before each of the measurements. Over the course of the measurements and review of the data, the number of preparation methods was reduced to a single method for reporting the concentration of each analyte, as shown in Table 2-1.

Table 2-1. Preparation Method Used in Reporting the Concentrations of Each of the Components of the Study Glasses

Component	Preparation Method
Al	PF
B	PF
Bi	LM
Ca	LM
Cr	LM
F	KH
Fe	PF
K	LM
Li	PF
Mn	PF
Na	LM
Ni	LM
P	LM
Pb	LM
Si	PF
W	LM

Samples of glass standards were also included in the analyses to provide an opportunity for checking the performance of the ICP-AES and IC instrumentation over the course of the analyses. Samples of Waste Compliance Plan Batch 1 glass (BCH)[7] were included in the ICP-AES portion of the analytical plan and samples of the low-level reference material (LRM)[8] were included in the IC portion of the analytical plan. The Batch 1 composition reported as the "Average Analyzed Glass Composition"[7] and the LRM composition reported as the "Consensus Average"[8] are used as the reference compositions of these glasses for the purposes of this study.

2.3.2 Viscosity Measurements

The viscosity of the as-received CJS-2018-2 glass sample was measured using a rotating spindle viscometer following the latest revision of SRNL Manual L29 Procedure ITS-0058.[9] A silicone viscosity standard fluid, traceable to the National Institute of Standards and Technology (NIST), was used to verify the operation of the viscometer at room temperature prior to use. The NIST 711 glass viscosity standard[10] was measured before and after the glass sample to determine the pre- and post-measurement spindle constants.

The temperature for the viscosity measurements began at 1150 °C, was increased to 1250 °C in 50 °C increments, reduced to 1150 °C, and then reduced to lower temperatures in 50 °C increments until the torque measurement was outside the range of the instrument. The glass was then heated back to 1150 °C for a final measurement. This hysteresis approach was intended to identify whether volatility or

[a] Samples prepared by AD were ultimately not used, since results for the PF and LM prepared samples indicated more complete dissolutions.

crystallization were influencing the results over the course of the measurement period. Three temperature and torque measurements were recorded at each incremental temperature, each separated by a period of five minutes. Viscosity (η) was calculated using the average of the pre- and post-measurement spindle constants (*K*) with the equation:[11]

$$\eta \text{ (Poise)} = K(T) \times (\text{percent (\%) torque}) / (\text{rotation speed})$$

where $K(T)$ is a linear equation for the spindle constant (K) as a function of temperature (T), which was determined using the equation above and the published viscosity values for the NIST traceable silicone viscosity standard fluid and NIST 711 glass viscosity standard. The averages of the three measurements were taken as the measured viscosity and temperature at a given incremental temperature. The resulting data were used to determine the viscosity-temperature relationship around the nominal melt temperature of 1150 °C. The average measured viscosity (η) data were fit with the Arrhenius equation:

$$\ln \eta = A + B \times 10^3/T$$

where A and B are constants and T is temperature in Kelvin. The linear fit information may be of value to PNNL in comparing the measured viscosity results among the laboratories participating in this round robin study.

2.3.3 Crystal Fraction Determination

The crystal fraction present after heat treatment at 950 °C was determined by X-ray diffraction (XRD). A mass of approximately 8 g of the as-received CJS-2018-2 glass was placed in a platinum-alloy crucible (Figure 2-1). A lid was fitted to the crucible and it was placed into a furnace preheated to 950 °C and held for 24 hours. The temperature of the furnace was recorded with an electronic data logger and verified to be stable throughout the heat treatment period. The crucible was then removed from the furnace and air quenched. The cooled glass was broken free of the crucible, photographed, and prepared XRD analysis.

Figure 2-1. Pieces of glass CJS-2018-2 in platinum crucible prior to heat treatment at 950 °C

To prepare for quantitative XRD analysis, the glass samples were first ground in a Spex 8000 mixer/mill to reduce the particle size and homogenize the samples. The ground glass powder was weighed and an internal standard, CaF_2, was added at 10% by mass. The ground glass powder and internal standard were reground with an agate mortar and pestle. The ground glass powder and standard mixture were packed into a 1 inch diameter, 1/16 inch deep well in a circular Plexiglas slide. The excess material on the surface was shaved off with a razor blade to reduce preferred orientation effects. Trevorite and nepheline standards were prepared from ultra-pure oxides. The trevorite and nepheline standards were each added to EA glass along with CaF_2 as an internal standard to make a series of 6, 8, and 10% by mass trevorite and nepheline standards. The mixtures were mounted in the same method on Plexiglas slides.

X-ray diffraction data were collected on a Bruker D8 X-ray Diffractometer[12] by step scanning over the 2Θ ranges of 20-70° with a step interval of 0.02° and a fixed counting time of 2 s per step. The instrument parameters are listed in Table 2-2. Search-match identification was performed with JADE software.[13]

Table 2-2. Instrument Parameters Used for Quantitative X-ray Diffraction

Radiation Source	CuKα X-ray
Source Power	45 kV, 40 mA
Wavelength	1.5405982 Å
Goniometer	Bruker D8
Divergence Slit	1º
Divergence Soller Slit	None
Divergence Antiscatter	1º
Specimen Rotation	No
Diffracted Beam Antiscatter	1º
Diffracted Beam Soller Slit	2º
Secondary Monochromator	Curved pyrolytic graphite
Receiving Slit	0.18º
Detector Slit	0.6º
Detector	NaI scintillation
2θ Range	20º - 70º
Step Interval	0.02º (2θ)
Fixed Counting Time	2 s/step

2.3.4 Heat Treatment

A portion of the as-received CJS-2018-2 glass sample was heat treated to simulate cooling along the centerline of a Waste Treatment and Immobilization Plant (WTP) HLW canister. This heat treatment is referred to as canister centerline cooled (CCC), and is described by Petkus.[14] A mass of about 80 g of glass pieces was placed in a platinum alloy crucible (Figure 2-2). A lid was fitted to the crucible, it was placed in a furnace preheated to 1050 °C, and the heat treatment schedule outlined in the experimental plan[3] was run via the furnace's programmable controller. The temperature of the furnace was recorded with an electronic data logger and was verified to have followed the schedule. At the end of the heat treatment schedule, the furnace was allowed to cool to less than 200 °C before removing the crucible. The cooled glass was broken free from the crucible, photographed, and prepared for XRD analysis in the manner described in Section 2.3.3.

Figure 2-2. Pieces of glass CJS-2018-2 in platinum crucible prior to the CCC heat treatment

2.3.5 Product Consistency Test

The as-received and CCC heat treated versions of glass CJS-2018-2 were subjected to the Product Consistency Test (PCT) Method A.[15] Four samples each of the as-received and CCC glasses were tested in triplicate. Also included were triplicate PCTs of the Approved Reference Material (ARM-1) glass,[7] triplicate PCTs of the Environmental Assessment (EA) glass,[16] and two reagent blank samples.

The glass samples were ground, sieved, washed, and prepared according to the standard procedure.[15] Fifteen milliliters of Type-I ASTM water were added to 1.5 g of glass in each stainless steel vessel. The vessels were closed, sealed, and placed in an oven at 90 ± 2 °C where they were maintained at temperature for 7 days. The vessels were then removed from the oven and allowed to cool to ambient temperature. Once cooled, an aliquot was drawn from each vessel and used to determine the ambient temperature pH of the leachate. Samples of the leachates were prepared by filtering and then diluting by adding 4 mL of 0.4 M HNO_3 to 6 mL of the leachate (a 6:10 volume to volume, v:v, dilution). The leachate samples for the EA glass were further diluted (1:10 v:v) with deionized water.

The prepared leachates were analyzed by ICP-AES under the auspices of an analytical plan.[3] Samples of a multi-element, standard solution[a] were also included in the analytical plan as a check on the accuracy of the ICP-AES instrument used for these measurements. Each sample was analyzed for concentrations of each of the following: aluminum (Al), boron (B), chromium (Cr), iron (Fe), lithium (Li), sodium (Na), and silicon (Si). Normalized concentrations were calculated based on the measured (as-received) compositions using the average of the common logarithms of the leachate concentrations.

2.4 <u>June 2019 Sample Characterization</u>

The viscosities of the as-received HLW-HCr-16-X(IV) and HLW-HCr-16-Y(VI) glass samples were measured in the manner described in Section 2.3.2. Heat treatments and crystal fraction determinations were completed in the manner described in Sections 2.3.3 and 2.3.4, with one exception: the mass of the

[a] ICP multi-element custom solution, product number SM-744-013, High Purity Standards, Charleston, SC

glasses for the CCC heat treatment was reduced from 80 g to 40 g since less sample was received from PNNL.

2.5 Additional Characterization of June 2014 Samples

The viscosities of the as-received HLW-HCr-16-X (II) and HLW-HCr-16-Y (I) glass samples were measured in the manner described in Section 2.3.2.

3.0 Results and Discussion

3.1 November 2018 Sample Characterization

3.1.1 Review and Evaluation of Chemical Composition Measurements

Table A-1, Table A-2, and Table A-3 in Appendix A provide the elemental concentration measurements in wt % for the CJS-2018-2 glass samples that were prepared by the PF, LM, and KH methods, respectively. Elemental measurements for samples of the Batch 1 and LRM reference glasses are also provided in these tables. These unprocessed data are provided as appendices to this report so that the values are readily available should they be of interest for future reviews.

In the sections that follow, the analytical sequences of the measurements are explored, the measurements of the standard glasses are investigated, the measurements for each glass are reviewed, and the average chemical composition for the glass is determined. JMP™ Pro Version 11.2.1 (SAS Institute, Inc.)[17] was used to support these analyses.

3.1.1.1 Treatment of Detection Limits

The elemental concentrations in Table A-1 through Table A-3 were converted to oxide concentrations by multiplying the values for each element by the gravimetric factor for the corresponding oxide. Some of the elemental concentration measurements were reported as being below the detection limit of the analytical processes used. In these cases, the value of the detection limit was considered to be the measured value of the analyte during the process of converting to oxide concentrations. This approach was used for the purposes of data review and calculating a sum of oxides for each glass. Those elements or oxides with one or more concentration measurements that were below the associated detection limit (BDL) will be denoted with a less than symbol (<) as the measured compositions are reported.

3.1.1.2 Measurements in Analytical Sequence

Exhibit A-1 in Appendix A provides plots of the wt % measurements generated for the study glasses by oxide and analytical block. The plots are in analytical sequence within each calibration block with different symbols and colors being used to represent each of the study and standard glasses. These plots include all the measurement data from Table A-1 through Table A-3, with each plotted point identified by its Lab ID. Plotting the data in this format provides an opportunity to identify gross trends in performance of the analytical instrument within and among calibration blocks. A review of these plots identified a slight, downward shift in the measurements for sodium in the second block. As will be shown in Section 3.1.1.4, the measured sodium values for all of the Batch 1 reference glass samples were within the acceptability limits, so this minor shift in sodium measurements is considered insignificant. There do not appear to be any other patterns or trends in the analytical process over the course of the composition measurements.

3.1.1.3 Composition Measurements by Glass Identifier

Exhibit A-2 in Appendix A provides plots of the oxide concentration measurements by the round robin ID (including the Batch 1 and LRM reference glasses), by Lab ID, and grouped by glass sub-sample. Different symbols and colors are used to represent the different glasses. These plots show the individual measurements across the duplicates of each preparation method and the two instrument calibrations for each glass. Plotting the data in this format provides an opportunity to review the values for each individual glass as a function of the duplicate preparations and duplicate measurements. A review of the plots presented in these exhibits reveals the repeatability of the four individual values for each oxide for each glass. No observations from Exhibit A-2 indicate an error in preparation or measurement. Therefore, the entire set of measurement data was used in determining representative, measured compositions for each sub-sample of the study glasses.

3.1.1.4 Results for the Batch 1 and LRM Reference Glasses

Exhibit A-3 in Appendix A provides a review of the Batch 1 and LRM results against acceptability limits.[18] The review is in the form of a plot of the measurements arranged by reference glass and element. The preparation method is indicated on each plot. The plotted measurements are framed by upper and lower acceptability limits for the element in question. The results show that all the measurements for the elements present in the Batch 1 and LRM reference glasses were within the acceptability limits.

3.1.1.5 Summary of the Measured Compositions

Given that no issues were identified with the measured values, all of the measurements for each oxide for each glass sample (i.e., all of the measurements in Appendix A, Table A-1 through Table A-3) were averaged to determine a representative chemical composition for each glass sample. The resulting values are provided in Table 3-1. A sum of oxides was also computed for each glass based upon the measured values. All the measured sums of oxides for the study glasses fall within the interval of 99.0 to 101.1 wt %, indicating acceptable recovery of all components.

Table 3-1. Measured Compositions of the CJS-2018-2 Glass Samples

Oxide (wt %)	CJS-2018-2 (1st sub-sample)	CJS-2018-2 (2nd sub-sample)	CJS-2018-2 (3rd sub-sample)	CJS-2018-2 (4th sub-sample)	Batch 1	LRM
Al_2O_3	18.9	18.9	19.0	18.7	4.48	-
B_2O_3	14.5	14.4	14.5	14.1	7.47	-
Bi_2O_3	0.782	0.765	0.766	0.761	<0.111	-
CaO	0.890	0.878	0.878	0.878	1.21	-
Cr_2O_3	1.82	1.86	1.90	1.91	<0.146	-
F	<0.05	<0.05	<0.05	<0.05	-	0.824
Fe_2O_3	5.55	5.62	5.69	5.56	12.3	-
K_2O	5.70	5.67	5.79	5.90	3.41	-
Li_2O	3.73	3.73	3.76	3.69	4.24	-
MnO	1.20	1.20	1.20	1.17	1.64	-
Na_2O	9.55	9.54	9.68	9.64	8.88	-
NiO	0.353	0.359	0.356	0.366	0.766	-
P_2O_5	0.387	0.385	0.376	0.388	<0.229	-
PbO	0.292	0.285	0.288	0.286	<0.108	-
SiO_2	36.3	35.8	36.6	35.5	50.8	-
WO_3	<0.13	<0.13	<0.13	<0.13	<0.126	-
Sum	100.1	99.6	101.0	99.0	95.9	0.8

3.1.2 Viscosity Measurements

Measured viscosity data for the as-received CJS-2018-2 glass sample are summarized in Table 3-2. The full set of measured data is provided in Table B-1 of Appendix B for reference. Details of the linear regression of the average measured viscosity data are shown in Exhibit B-1 of Appendix B. The R^2 value of about 0.99 demonstrates that there is no appreciable difference between the measured and fitted values. No hysteresis in the measured values is apparent, indicating that neither crystallization nor volatility influenced the measurements.

Table 3-2. Measured Viscosity Data for Glass CJS-2018-2

Temperature (°C)	Average Measured Viscosity (Poise)
1156	49
1203	38
1253	27
1153	59
1106	86
1052	140
1006	228
956	438
903	912
856	1683
802	4143
751	11,269
1157	52

3.1.3 Crystal Fraction Determinations

The fractions of crystalline material present in glass CJS-2018-2 after heat treatments were determined by XRD. After heat treatment at 950 °C for 24 hours, minor crystallization was visible on the cooled glass along the edge in contact with the crucible (Figure 3-1). A larger amount of crystallization was visible on the cooled glass after the CCC heat treatment (Figure 3-2).

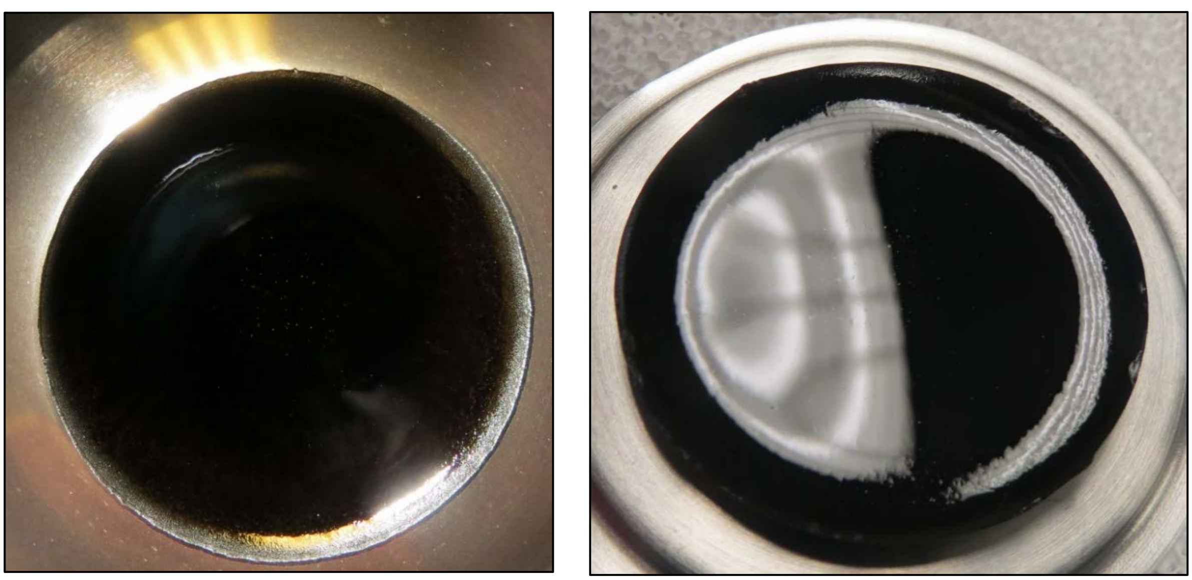

Figure 3-1. Glass CJS-2018-2 after heat treatment at 950 °C for 24 hours showing crystallization along the edge in contact with the crucible, before (left) and after (right) removal from the crucible

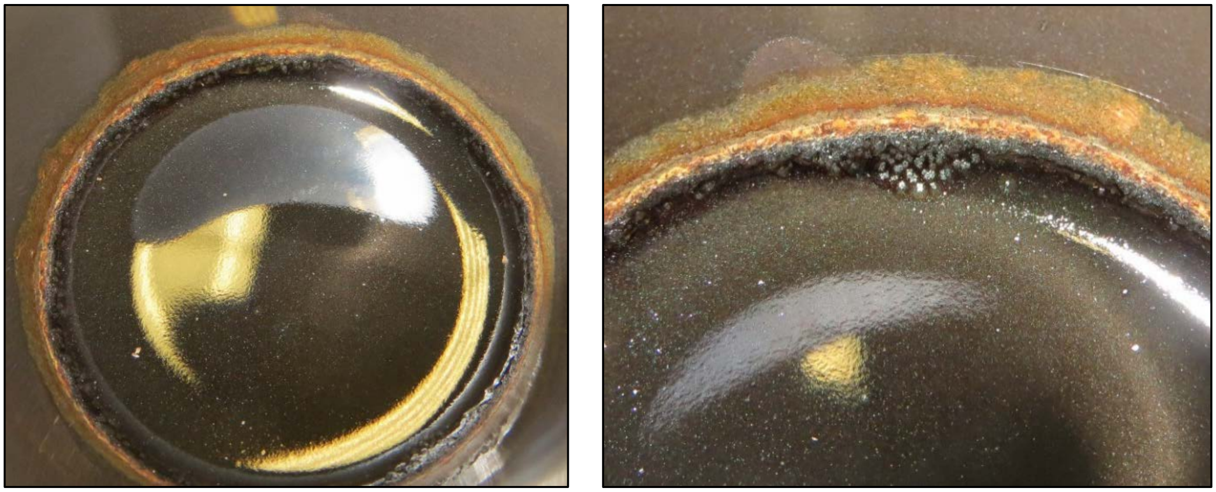

Figure 3-2. Glass CJS-2018-2 after the CCC heat treatment showing crystallization across the top surface (left) and at the interface with the crucible (right)

Trevorite was identified in each of the glasses by XRD, as shown in Table 3-3. The XRD spectra for the glasses heat treated at 950 °C for 24 hours and via the CCC heat treatment schedule are shown in Figure 3-3 and Figure 3-4, respectively.

Table 3-3. Crystal Fraction Measurements for Glass CJS-2018-2

Heat Treatment	Crystal Fraction
950 °C, 24 hours	Trevorite, 4.5 wt %
WTP HLW CCC	Trevorite, 7.1 wt %

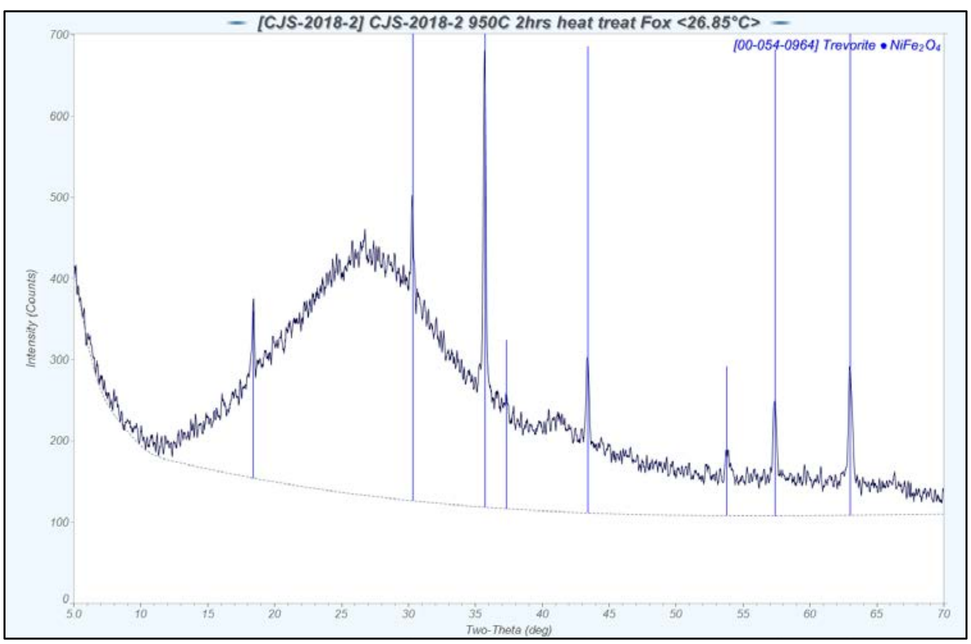

Figure 3-3. XRD spectrum for glass CJS-2018-2 after heat treatment at 950 °C for 24 hours

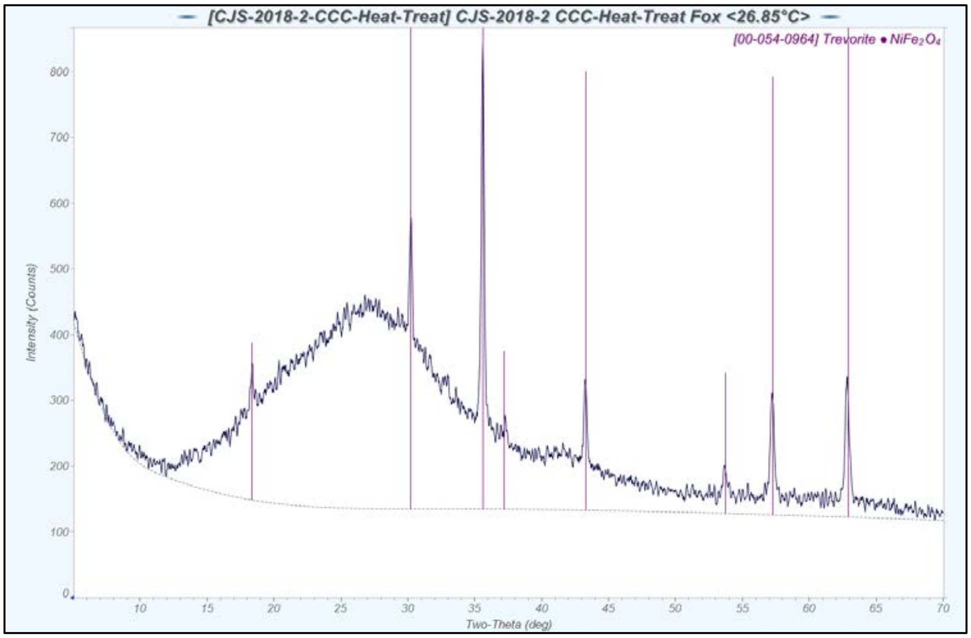

Figure 3-4. XRD spectrum for glass CJS-2018-2 after the CCC heat treatment

3.1.4 Review and Evaluation of PCT Measurements

Table C-1 in Appendix C provides the elemental leachate concentration measurements for the solution samples generated by the PCTs for the study glasses and standards. The values for these measurements are given in the table as-received ("ar") from the laboratory analyses. The values after adjustments for the dilution factors are given in Table C-2 in Appendix C. The measurements for the study glasses, blanks, and the ARM-1 glass were multiplied by 1.6667 to determine the values in mg/L.

Based on the masses of the PCT vessels before and after the 7-day procedures, there were no water-loss issues. The ratio of leachant volume to the mass of ground glass was confirmed to be correct for each vessel. The measured concentrations of B, Li, Na, and Si in the leachates from the ARM-1 glasses fell within the control charts for these results,[7] with the exception of one Si measurement. The expectation is that an error in the performance of a PCT would result in a consistent divergence of the concentrations of the analytes of the ARM-1 glass away from the limits of the control charts. Since there were no consistent issues with the ARM values for the PCTs, the tests were considered to have been performed properly and no bias correction was performed.[a]

The measured, ambient temperature pH values for each of the PCT leachates are provided in Table C-3 of Appendix C for reference.

In the sections that follow, the analytical sequences of the measurements are explored, the measurements for each glass are reviewed, the measurements of the multi-element solution standard are investigated, the normalized PCT results for each glass sample are determined, and comparisons are made between the PCT results for the two heat treatments of the round robin glass. JMP Pro Version 11.2.1 (SAS Institute, Inc.)[17] was used to support these analyses.

[a] Data are provided in Appendix B to support bias correction per ASTM C 1285, Section 25.2 if desired.

3.1.4.1 Treatment of Detection Limits

Some of the "ar" measurements (Table C-1 in Appendix C) were below the analytical detection limits. These measurements (indicated by a "<" symbol in Table C-1) were replaced by their detection limits in subsequent analyses for the purposes of data review and of calculating normalized leachate values. Those elements with measured concentrations that were below the associated detection limit will be denoted with a less than symbol (<) as the normalized leachate values are reported.

3.1.4.2 Results for the Samples of the Multi-Element Solution Standard

Table 3-4 provides a review of the measurements of the solution standard samples that were included in the analytical blocks for the PCT analyses. For each analytical block, the mean, standard deviation, and percent relative standard deviation (%RSD) are determined for each element present in the standard. Following the guidance in ASTM C 1285, there were two primary evaluations conducted for these summary statistics: the mean value for each analytical block was found to be less than 10% from the reference value (i.e., a percent relative bias less than 10%) for the element in question, and the %RSD was less than 10% for the element in question. The results in Table 3-4 satisfy these criteria, and thus, the results for the solution standard suggest no significant issues with the analytical outcomes for the measurements of the PCT solutions.

Table 3-4. Results from Samples of the Multi-Element Solution Standard

Oven Run	**1**			**Reference Values (mg/L)**
Block	**1**	**2**	**3**	
Mean (B (mg/L))	20.00	20.62	20.17	20
Mean (Li (mg/L))	10.06	10.34	10.11	10
Mean (Na (mg/L))	81.97	80.20	81.57	81
Mean (Si (mg/L))	47.54	49.87	49.96	50
% relative bias B	0.0%	3.1%	0.8%	<10% per ASTM C 1285
% relative bias Li	0.6%	3.4%	1.1%	
% relative bias Na	1.2%	-1.0%	0.7%	
% relative bias Si	-4.9%	-0.3%	-0.1%	
Std Dev (B (mg/L))	0.946	0.198	0.303	
Std Dev (Li (mg/L))	0.069	0.009	0.059	
Std Dev (Na (mg/L))	0.505	0.478	0.472	
Std Dev (Si (mg/L))	2.392	0.236	0.491	
%RSD (B (mg/L))	4.7%	1.0%	1.5%	<10% per ASTM C 1285
%RSD (Li (mg/L))	0.7%	0.1%	0.6%	
%RSD (Na (mg/L))	0.6%	0.6%	0.6%	
%RSD (Si (mg/L))	5.0%	0.5%	1.0%	

3.1.4.3 Measurements in Analytical Sequence

Exhibit C-1 in Appendix C provides plots of the common logarithms of the leachate concentrations (mg/L) in analytical sequence by analytical block. Plotting the data in this format provides an opportunity to identify gross trends in performance of the analytical instrument within and among calibration blocks. No issues were observed in a review of these plots.

3.1.4.4 Measurements by Glass Identifier

Exhibit C-2 in Appendix C provides plots of the leachate concentrations for both the quenched and CCC versions of each of the study glasses and for the standards for each analytical set. These plots are in common logarithms of the mg/L values and allow for the assessment of the repeatability of the measurements for each glass. For some of the glasses, minor scatter among the triplicate values of some analytes is observed. A closer look at the quenched and CCC outcomes is provided in the following sections.

3.1.4.5 Normalization of the PCT Results

The PCT leachate data were used to determine normalized concentrations for each element of interest using the measured (as-received) composition of the glasses. The average of the four subsample compositions shown in Table 3-1 was taken as the measured composition of the CJS-2018-2 round robin glass for the purpose of calculating normalized concentrations. The normalized concentrations for each element of interest in the PCT were calculated following the expression given in ASTM C1285:

$$NC_i = \frac{c_i\left(sample\right)}{f_i}$$

where NC_i is the normalized concentration in units of $g_{\text{waste form}}/L_{\text{leachant}}$, c_i is the concentration of element "*i*" in the leachate in units of g_i/L, and f_i is the mass fraction of element "*i*" in the unleached glass in units of g_i/g_{glass}.[a]

An equation was developed to allow for calculation of the NC_i values using the units of measurement provided with the analytical results for this study, and to accommodate the triplicate leachate measurements for each of the study glasses. Note that the symbols in this second equation were kept consistent with those used in ASTM C1285, but the units of measurement differ. The common logarithm of the normalized concentration for each element "*i*" (NC_i) for each of the glass subsamples was determined using the equation:

$$\log_{10}\left(NC_i\right) = \overline{\log_{10} c_i} - \left[1 + \log_{10} f_i\right]$$

where NC_i remains in units of $g_{\text{waste form}}/L_{\text{leachant}}$, $\overline{\log_{10} c_i}$ is the average of the common logarithms of the measured concentrations of element "*i*" in the triplicate leachates in units of mg/L, and $\log_{10} f_i$ is the common logarithm of the measured concentration of element "*i*" in the glass in units of wt %. The calculated NC_i values are discussed further in the following sections.

3.1.4.6 Effects of Heat Treatments

Exhibit C-3 in Appendix C provides plots of the normalized PCT responses for the two heat treatments for the glass subsamples as well as the responses for ARM-1 and EA. Note that an indicator is provided as part of these plots to show results involving BDL values. The plots of Exhibit C-3 provide a graphical comparison between the PCT responses for the two heat treatments of each glass subsample. Table 3-5 provides a listing of the normalized PCT responses. In general, heat treatment appears to have had little impact on the PCT responses among the glass subsamples.

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[a] Note that the waste forms in this study were assumed to be of similar density. The PCT-A reference volume of leachant to sample mass ratio was used, and the 100 to 200 mesh reference particle size was used. Thus, no adjustment for the density of the glasses was made in normalizing the PCT results. Data provided in the appendices of this report allow for the calculation of normalized elemental mass loss (NL_i) if glass densities are measured at a later date.

Table 3-5. PCT Results, Normalized to Measured Composition

Glass ID	Heat Treatment	NC_{Al} (g/L)	NC_B (g/L)	NC_{Cr} (g/L)	NC_{Fe} (g/L)	NC_{Li} (g/L)	NC_{Na} (g/L)	NC_{Si} (g/L)
ARM-1	ref	0.224	0.418	NA	NA	0.516	0.471	0.234
EA	ref	<0.851	15.797	NA	<0.265	9.041	12.568	3.649
CJS-2018-2 (1st sub-sample)	as received	0.333	1.338	<0.130	0.163	1.295	0.944	0.285
CJS-2018-2 (2nd sub-sample)	as received	0.329	1.111	<0.130	0.171	1.109	0.859	0.281
CJS-2018-2 (3rd sub-sample)	as received	0.324	1.247	<0.130	0.158	1.218	0.886	0.279
CJS-2018-2 (4th sub-sample)	as received	0.331	1.207	<0.130	0.168	1.190	0.894	0.281
CJS-2018-2 (1st sub-sample)	ccc	0.243	2.208	<0.130	0.077	2.264	1.114	0.293
CJS-2018-2 (2nd sub-sample)	ccc	0.295	1.322	<0.130	0.104	1.286	0.855	0.268
CJS-2018-2 (3rd sub-sample)	ccc	0.306	1.288	<0.130	0.111	1.237	0.861	0.271
CJS-2018-2 (4th sub-sample)	ccc	0.313	1.303	<0.130	0.119	1.263	0.899	0.278

3.2 June 2019 Sample Characterization

3.2.1 Viscosity Measurements

Measured viscosity data for as-received samples of glasses HLW-HCr-16-X(IV) and HLW-HCr-16-Y(VI) are summarized in Table 3-6. The full sets of measured data are provided in Table B-2 and Table B-3 of Appendix B for reference. Details of the linear regressions of the average measured viscosity data are shown in Exhibit B-2 and Exhibit B-3 of Appendix B. The R^2 values of about 0.99 demonstrate that there are no appreciable differences between the measured and fitted values. No hysteresis in the measured values is apparent, indicating that neither crystallization nor volatility influenced the measurements.

Table 3-6. Measured Viscosity Data for Glasses HLW-HCr-16-X(IV) and HLW-HCr-16-Y(VI)

HLW-HCr-16-X(IV)			HLW-HCr-16-Y(VI)	
Temperature (°C)	Average Measured Viscosity (Poise)		Temperature (°C)	Average Measured Viscosity (Poise)
1150	59		1149	64
1200	43		1200	46
1250	32		1250	33
1150	67		1149	70
1100	100		1100	105
1050	158		1050	165
1000	258		1000	271
950	448		950	465
900	824		900	875
850	1652		850	1753
800	3897		801	4147
750	10,246		750	11,587
1150	67		1150	67

3.2.2 Crystal Fraction Determinations

The fractions of crystalline material present in glasses HLW-HCr-16-X(IV) and HLW-HCr-16-Y(VI) after heat treatments were determined by XRD. After heat treatment at 950 °C for 24 hours, minor crystallization was visible on the cooled glasses along the edges in contact with the crucibles (Figure 3-5). A larger amount of crystallization was visible on the surfaces of the cooled glasses after the CCC heat treatment (Figure 3-6).

HLW-HCr-16-X(IV)
(CJS-2018_1)

HLW-HCr-16-Y(VI)
(CJS-2018_2)

Figure 3-5. Minor crystallization visible on the top surfaces of the glasses after heat treatment at 950 °C

HLW-HCr-16-X(IV)
(CJS-2018_1)

HLW-HCr-16-Y(VI)
(CJS-2018_2)

Figure 3-6. Heavy crystallization visible on the top surfaces of the glasses after the CCC heat treatment

Trevorite was identified in each of the glasses by XRD, as shown in Table 3-7. The amount of trevorite present in each of the glasses after each of the heat treatments is similar to the amounts shown earlier in Table 3-3 for Glass CJS-2018-2. Note, however, that glass HLW-HCr-16-X(IV) also contains nepheline after the CCC heat treatment. The XRD spectra for glass HLW-HCr-16-X(IV) for the two heat treatments are shown in Figure 3-7 and Figure 3-8. The XRD spectra for glass HLW-HCr-16-Y(VI) for the two heat treatments are shown in Figure 3-9 and Figure 3-10.

Table 3-7. Crystal Fraction Measurements for Glasses HLW-HCr-16-X(IV) and HLW-HCr-16-Y(VI)

Glass ID	Heat Treatment	Crystal Fraction
HLW-HCr-16-X(IV) (CJS-2018_1)	950 °C, 24 hours	Trevorite, 5.0 wt %
	WTP HLW CCC	Trevorite, 7.6 wt % Nepheline, 5.2 wt %
HLW-HCr-16-Y(VI) (CJS-2018_2)	950 °C, 24 hours	Trevorite, 5.3 wt %
	WTP HLW CCC	Trevorite, 7.8 wt %

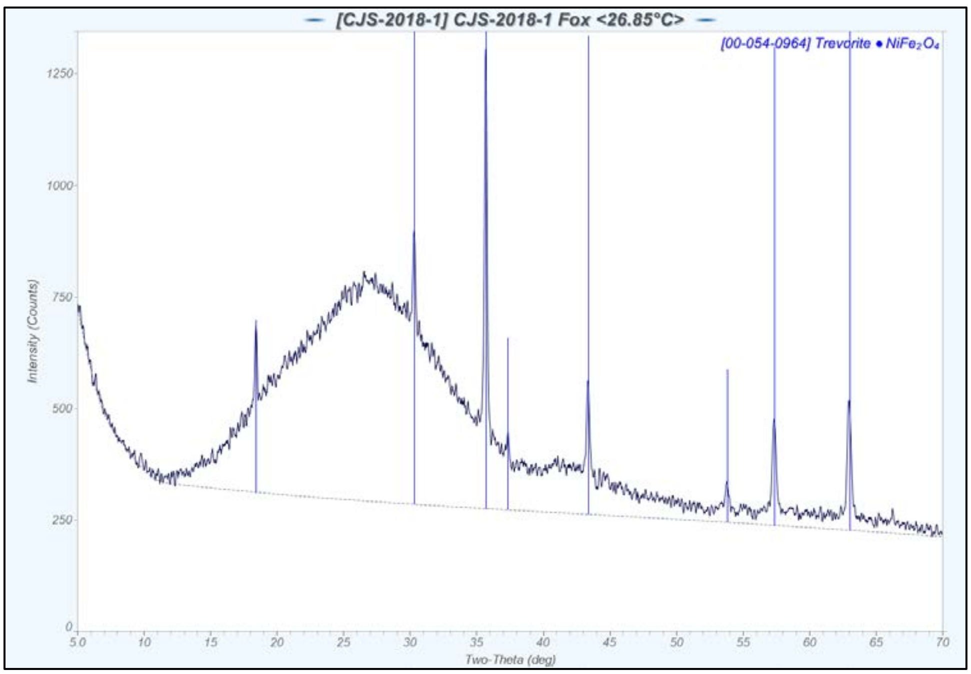

Figure 3-7. XRD spectrum for glass HLW-HCr-16-X(IV) after heat treatment at 950 °C for 24 hours

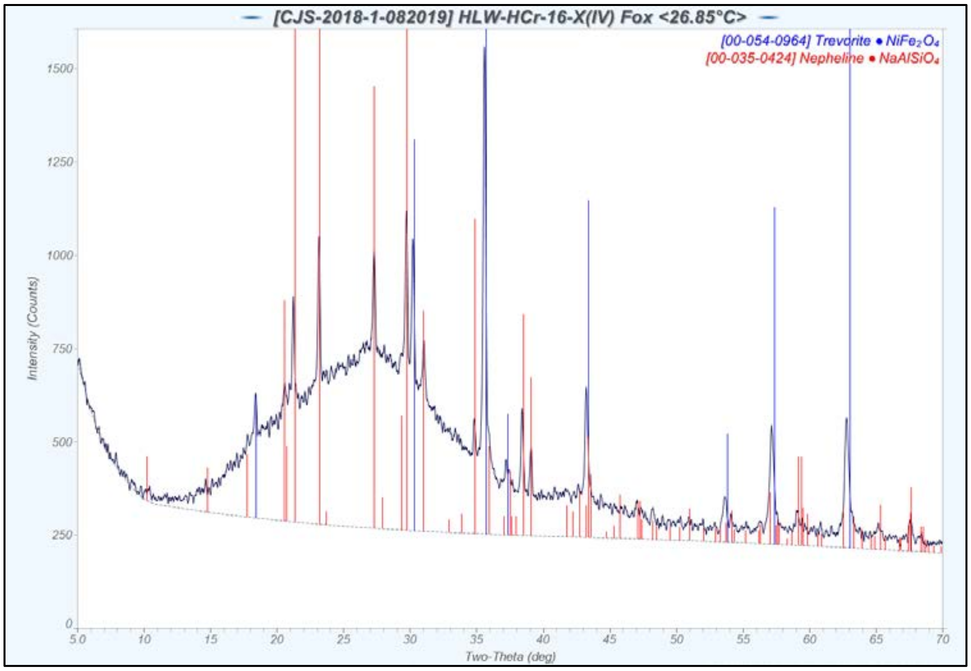

Figure 3-8. XRD spectrum for glass HLW-HCr-16-X(IV) after the CCC heat treatment

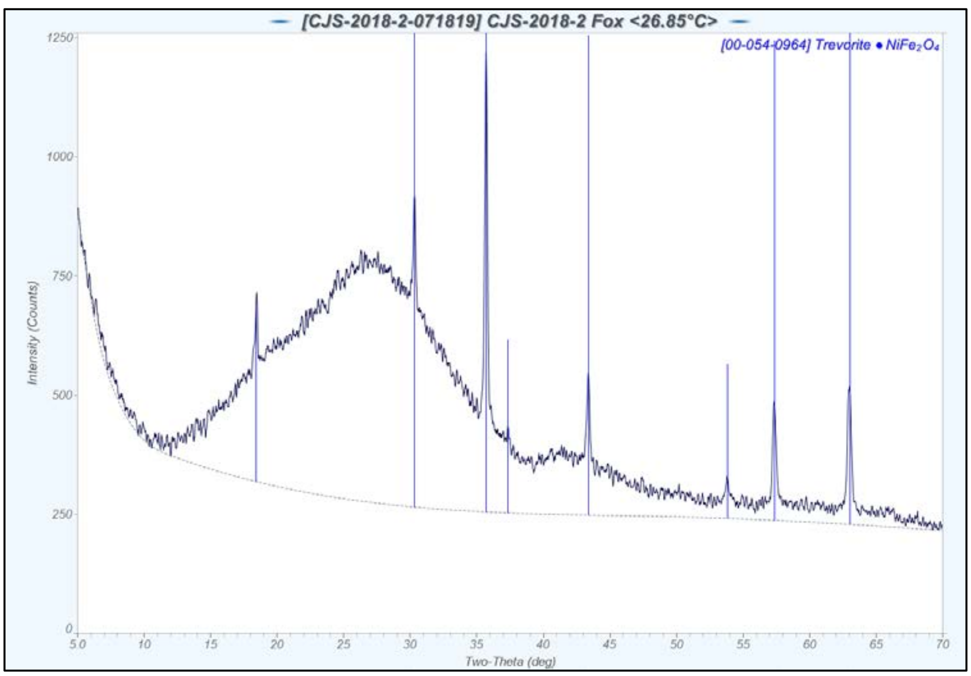

Figure 3-9. XRD spectrum for glass HLW-HCr-16-Y(VI) after heat treatment at 950 °C for 24 hours

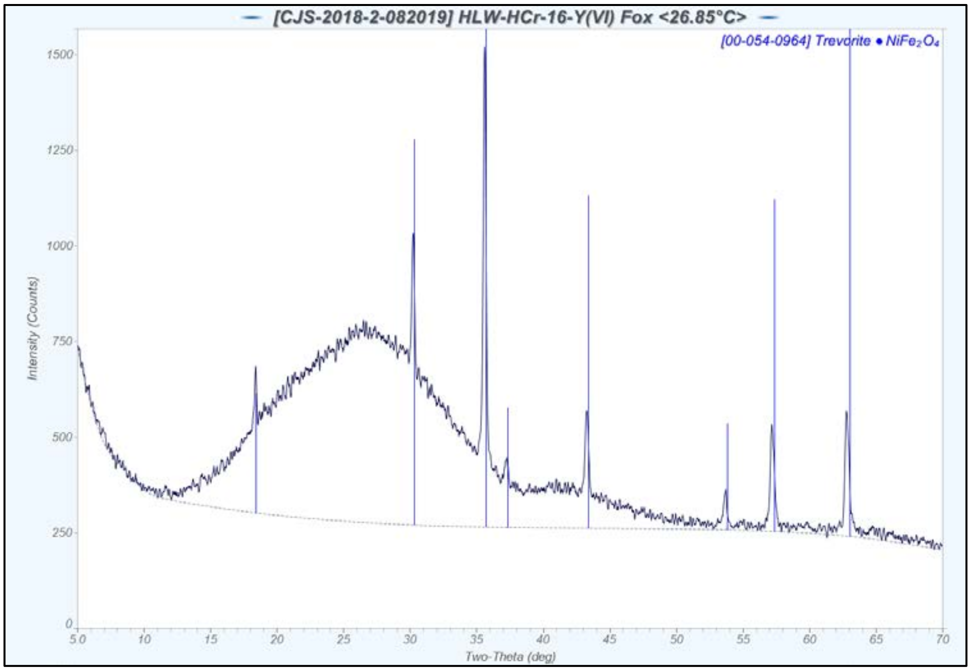

Figure 3-10. XRD spectrum for glass HLW-HCr-16-Y(VI) after the CCC heat treatment

3.3 Additional Characterization of June 2014 Samples

Measured viscosity data for as-received samples of glasses HLW-HCr-16-Y(I) and HLW-HCr-16-X(II) are summarized in Table 3-8. The full sets of measured data are provided in Table B-4 and Table B-5 of Appendix B for reference. Details of the linear regressions of the average measured viscosity data are shown in Exhibit B-4 and Exhibit B-5 of Appendix B. The R^2 values of about 0.99 demonstrate that there are no appreciable differences between the measured and fitted values. No hysteresis in the measured values is apparent, indicating that neither crystallization nor volatility influenced the measurements.

Table 3-8. Measured Viscosity Data for Glasses HLW-HCr-16-Y(I) and HLW-HCr-16-X(II)

HLW-HCr-16-Y(I)			HLW-HCr-16-X(II)	
Temperature (°C)	Average Measured Viscosity (Poise)		Temperature (°C)	Average Measured Viscosity (Poise)
1150	70		1150	61
1200	51		1200	44
1250	38		1250	32
1150	79		1150	68
1100	118		1100	103
1050	179		1050	161
1000	289		1000	264
950	486		950	458
900	873		901	835
850	1735		851	1717
800	3773		800	3995
750	9658		750	11083
1150	75		1150	64

4.0 Summary

This report presents the results of glass characterization performed at SRNL in support of a DOE-ORP round robin study of simulated high-level waste glasses produced in small scale melters. The round robin study is coordinated by PNNL. SRNL received a series of glass samples from PNNL as part of this study, including CJS-2018-2, HLW-HCr-16-X(IV) [also labeled CJS-2018_1], HLW-HCr-16-Y(VI) [also labeled CJS-2018_2], HLW-HCr-16-X (II), and HLW-HCr-16-Y (I).

SRNL performed chemical composition measurements, heat treatments, crystal fraction measurements via XRD, viscosity determinations, and chemical durability evaluations using the ASTM PCT. The results of this work are detailed in this report. PNNL will use these data for comparisons with the results provided by other laboratories participating in the round robin study.

5.0 References

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Appendix A Tables and Exhibits Supporting the Glass Composition Measurements

Table A-1. PF Measurements of the CJS-2018-2 Glass Samples

Glass ID	Block	Sequence	Lab ID	Al (wt %)	B (wt %)	Fe (wt %)	Li (wt %)	Mn (wt %)	Si (wt %)
Batch 1	1	1	BCHPF11	2.35	2.36	8.57	1.97	1.26	21.6
CJS-2018-2 (4th sub-sample)	1	2	S-8565PF21	10.0	4.47	3.97	1.73	0.922	17.2
CJS-2018-2 (3rd sub-sample)	1	3	S-8564PF11	10.1	4.49	3.99	1.73	0.925	17.2
CJS-2018-2 (4th sub-sample)	1	4	S-8565PF11	9.87	4.40	3.91	1.71	0.909	17.0
CJS-2018-2 (1st sub-sample)	1	5	S-8562PF21	10.0	4.58	3.96	1.74	0.940	17.4
Batch 1	1	6	BCHPF12	2.38	2.35	8.63	1.97	1.27	25.4
CJS-2018-2 (1st sub-sample)	1	7	S-8562PF11	9.85	4.43	3.84	1.71	0.913	17.1
CJS-2018-2 (2nd sub-sample)	1	8	S-8563PF21	9.95	4.44	3.91	1.72	0.916	16.9
CJS-2018-2 (3rd sub-sample)	1	9	S-8564PF21	10.2	4.63	4.07	1.77	0.950	17.6
CJS-2018-2 (2nd sub-sample)	1	10	S-8563PF11	10.2	4.58	4.04	1.76	0.945	17.2
Batch 1	1	11	BCHPF13	2.36	2.31	8.64	1.97	1.27	21.9
Batch 1	2	1	BCHPF21	2.37	2.30	8.58	1.97	1.27	24.8
CJS-2018-2 (1st sub-sample)	2	2	S-8562PF12	10.0	4.43	3.85	1.74	0.931	17.3
CJS-2018-2 (2nd sub-sample)	2	3	S-8563PF12	9.89	4.43	3.90	1.74	0.923	16.4
CJS-2018-2 (3rd sub-sample)	2	4	S-8564PF22	10.1	4.48	3.97	1.75	0.934	16.8
CJS-2018-2 (3rd sub-sample)	2	5	S-8564PF12	9.82	4.42	3.91	1.73	0.920	16.7
Batch 1	2	6	BCHPF22	2.37	2.26	8.50	1.95	1.25	24.6
CJS-2018-2 (2nd sub-sample)	2	7	S-8563PF22	9.98	4.44	3.88	1.73	0.921	16.5
CJS-2018-2 (4th sub-sample)	2	8	S-8565PF12	9.84	4.40	3.85	1.73	0.907	16.4
CJS-2018-2 (4th sub-sample)	2	9	S-8565PF22	9.82	4.29	3.82	1.69	0.901	15.8
CJS-2018-2 (1st sub-sample)	2	10	S-8562PF22	10.1	4.56	3.88	1.74	0.940	15.9
Batch 1	2	11	BCHPF23	2.41	2.34	8.74	2.00	1.29	24.3

Table A-2. LM Measurements of the CJS-2018-2 Glass Samples

Glass ID	Block	Sequence	Lab ID	Bi (wt %)	Ca (wt %)	Cr (wt %)	K (wt %)	Na (wt %)	Ni (wt %)	P (wt %)	Pb (wt %)	W (wt %)
Batch 1	1	1	BCHLM11	<0.100	0.854	<0.100	2.79	6.57	0.592	<0.100	<0.100	<0.100
CJS-2018-2 (4th sub-sample)	1	2	S-8565LM21	0.660	0.610	1.31	5.10	7.52	0.277	0.169	0.257	<0.100
CJS-2018-2 (3rd sub-sample)	1	3	S-8564LM11	0.685	0.623	1.31	4.86	7.58	0.274	0.161	0.266	<0.100
CJS-2018-2 (3rd sub-sample)	1	4	S-8564LM21	0.682	0.624	1.28	4.74	7.43	0.277	0.161	0.267	<0.100
CJS-2018-2 (4th sub-sample)	1	5	S-8565LM11	0.681	0.622	1.29	4.83	7.42	0.285	0.159	0.263	<0.100
Batch 1	1	6	BCHLM12	<0.100	0.862	<0.100	2.83	6.74	0.605	<0.100	<0.100	<0.100
CJS-2018-2 (2nd sub-sample)	1	7	S-8563LM21	0.672	0.610	1.25	4.66	7.24	0.275	0.164	0.258	<0.100
CJS-2018-2 (1st sub-sample)	1	8	S-8562LM11	0.702	0.634	1.22	4.63	7.28	0.277	0.169	0.270	<0.100
CJS-2018-2 (2nd sub-sample)	1	9	S-8563LM11	0.695	0.635	1.23	4.56	7.17	0.284	0.170	0.267	<0.100
CJS-2018-2 (1st sub-sample)	1	10	S-8562LM21	0.702	0.637	1.19	4.63	7.11	0.276	0.171	0.271	<0.100
Batch 1	1	11	BCHLM13	<0.100	0.877	<0.100	2.86	6.81	0.617	<0.100	<0.100	<0.100
Batch 1	2	1	BCHLM21	<0.100	0.860	<0.100	2.76	6.52	0.603	<0.100	<0.100	<0.100
CJS-2018-2 (1st sub-sample)	2	2	S-8562LM22	0.694	0.630	1.26	4.79	7.00	0.275	0.166	0.265	<0.100
CJS-2018-2 (2nd sub-sample)	2	3	S-8563LM12	0.680	0.622	1.26	4.72	7.01	0.279	0.166	0.259	<0.100
CJS-2018-2 (3rd sub-sample)	2	4	S-8564LM22	0.671	0.614	1.28	4.79	6.83	0.274	0.160	0.258	<0.100
CJS-2018-2 (4th sub-sample)	2	5	S-8565LM12	0.689	0.634	1.27	4.57	6.74	0.293	0.167	0.271	<0.100
Batch 1	2	6	BCHLM22	<0.100	0.864	<0.100	2.92	6.38	0.600	<0.100	<0.100	<0.100
CJS-2018-2 (1st sub-sample)	2	7	S-8562LM12	0.707	0.644	1.30	4.87	6.95	0.283	0.170	0.278	<0.100
CJS-2018-2 (2nd sub-sample)	2	8	S-8563LM22	0.697	0.641	1.36	4.88	6.90	0.291	0.173	0.274	<0.100
CJS-2018-2 (4th sub-sample)	2	9	S-8565LM22	0.700	0.646	1.35	5.08	6.93	0.298	0.183	0.272	<0.100
CJS-2018-2 (3rd sub-sample)	2	10	S-8564LM12	0.711	0.649	1.32	4.84	6.88	0.294	0.174	0.277	<0.100
Batch 1	2	11	BCHLM23	<0.100	0.867	<0.100	2.82	6.49	0.598	<0.100	<0.100	<0.100

Table A-3. KH Measurements of the CJS-2018-2 Glass Samples

Glass ID	Block	Sequence	Lab ID	F (wt %)
LRM	1	1	LRMKH11	0.838
CJS-2018-2 (1st sub-sample)	1	2	S-8562KH21	<0.050
CJS-2018-2 (2nd sub-sample)	1	3	S-8563KH11	<0.050
CJS-2018-2 (3rd sub-sample)	1	4	S-8564KH21	<0.050
CJS-2018-2 (4th sub-sample)	1	5	S-8565KH21	<0.050
LRM	1	6	LRMKH12	0.813
CJS-2018-2 (4th sub-sample)	1	7	S-8565KH11	<0.050
CJS-2018-2 (2nd sub-sample)	1	8	S-8563KH21	<0.050
CJS-2018-2 (1st sub-sample)	1	9	S-8562KH11	<0.050
CJS-2018-2 (3rd sub-sample)	1	10	S-8564KH11	<0.050
LRM	1	11	LRMKH13	0.829
LRM	2	1	LRMKH21	0.828
CJS-2018-2 (4th sub-sample)	2	2	S-8565KH22	<0.050
CJS-2018-2 (4th sub-sample)	2	3	S-8565KH12	<0.050
CJS-2018-2 (2nd sub-sample)	2	4	S-8563KH12	<0.050
CJS-2018-2 (1st sub-sample)	2	5	S-8562KH12	<0.050
LRM	2	6	LRMKH22	0.827
CJS-2018-2 (3rd sub-sample)	2	7	S-8564KH22	<0.050
CJS-2018-2 (1st sub-sample)	2	8	S-8562KH22	<0.050
CJS-2018-2 (2nd sub-sample)	2	9	S-8563KH22	<0.050
CJS-2018-2 (3rd sub-sample)	2	10	S-8564KH12	<0.050
LRM	2	11	LRMKH23	0.806

Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence

Analyte=Al2O3 (wt%), Prep Method=PF
Variability Chart for Measured

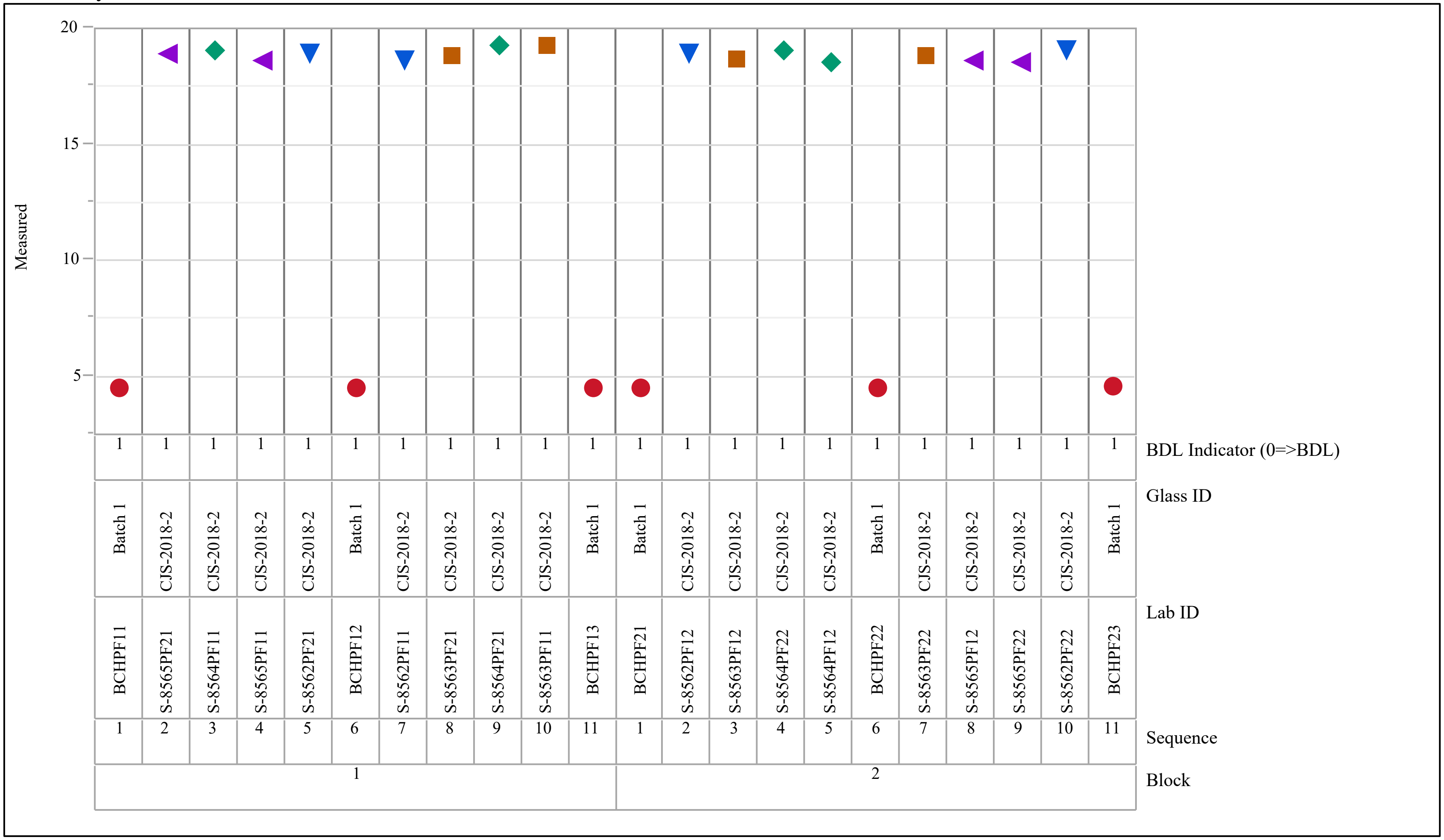


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=B2O3 (wt%), Prep Method=PF
Variability Chart for Measured

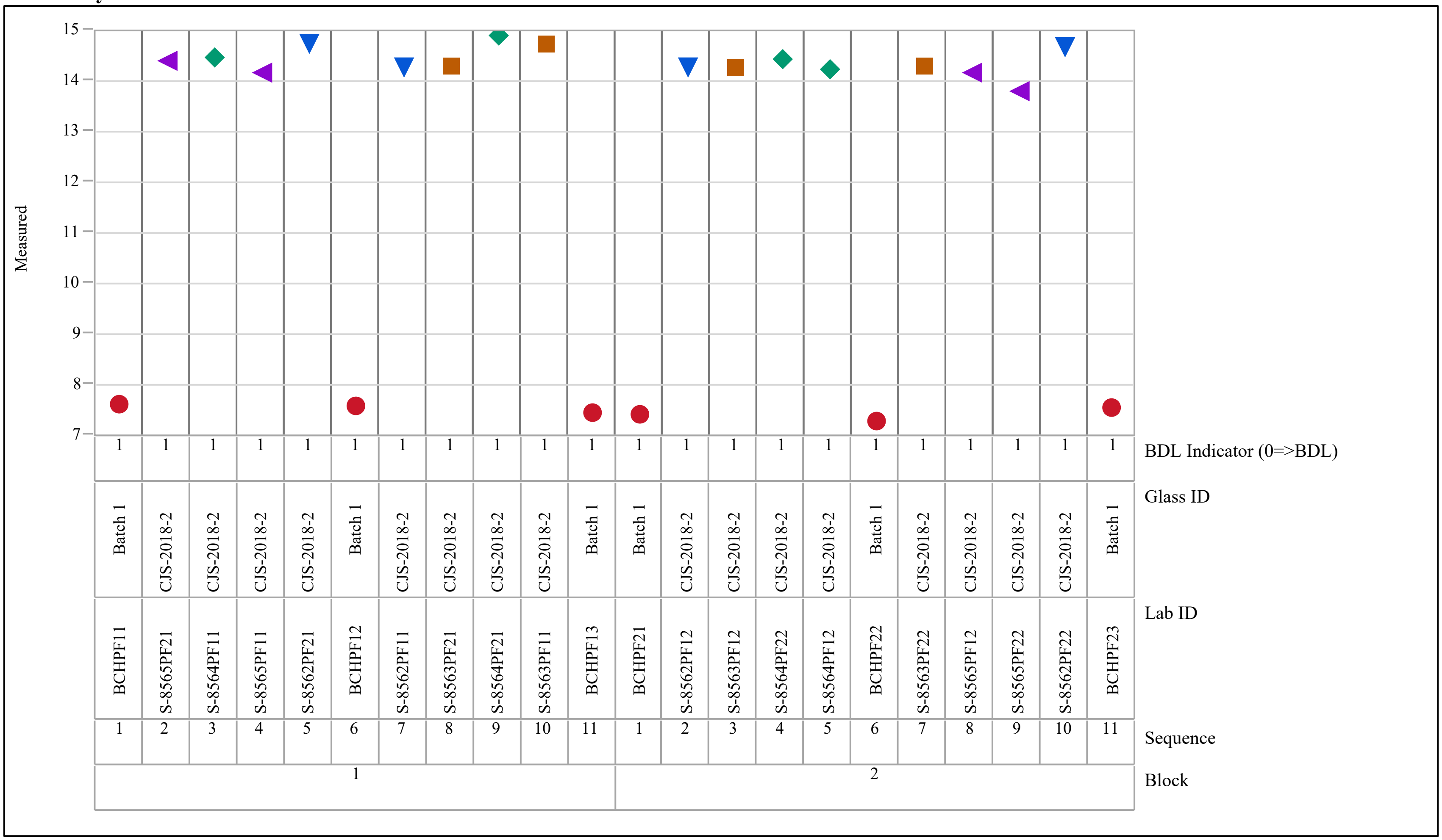


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=Bi2O3 (wt%), Prep Method=LM
Variability Chart for Measured

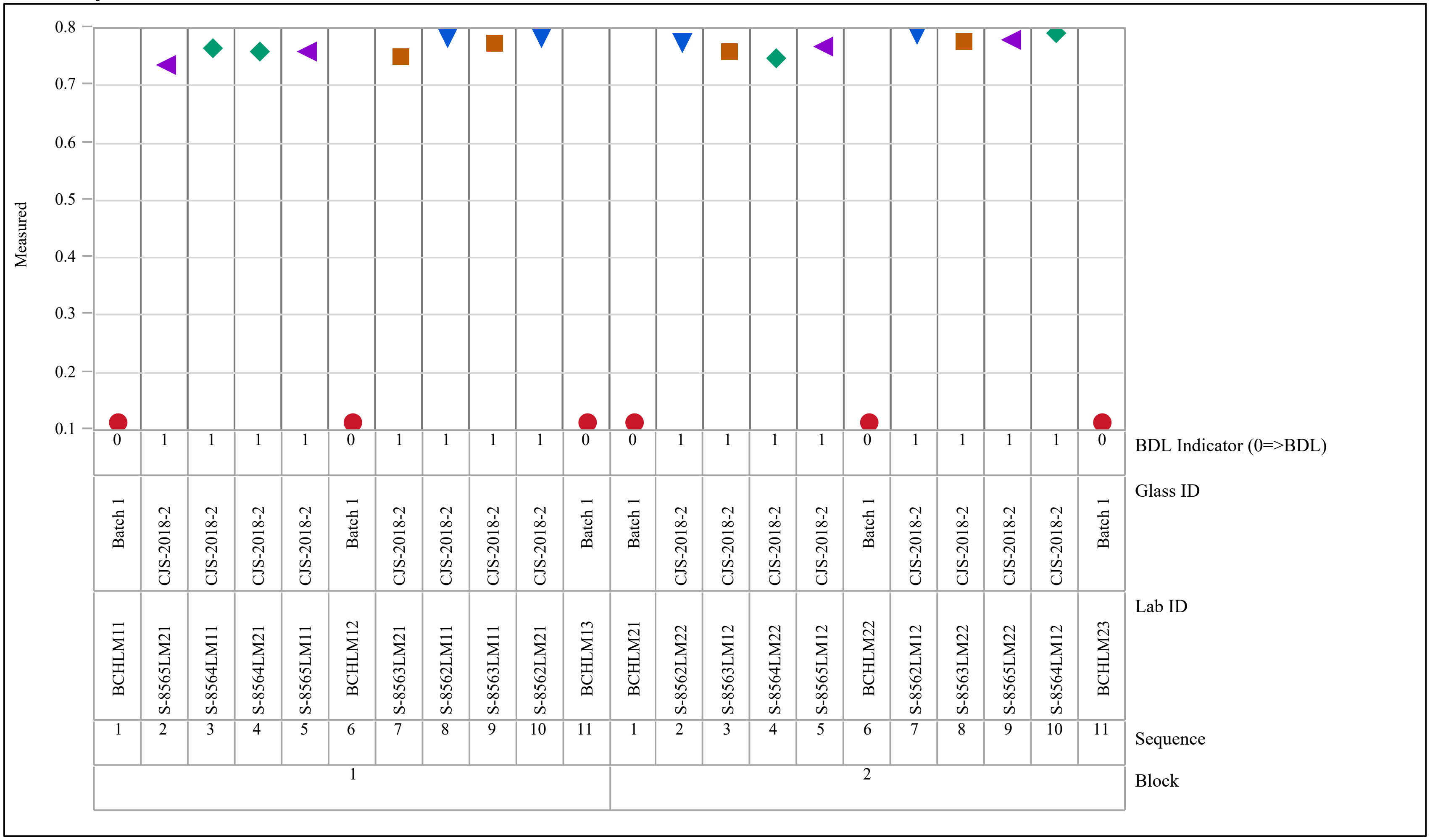


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=CaO (wt%), Prep Method=LM
Variability Chart for Measured

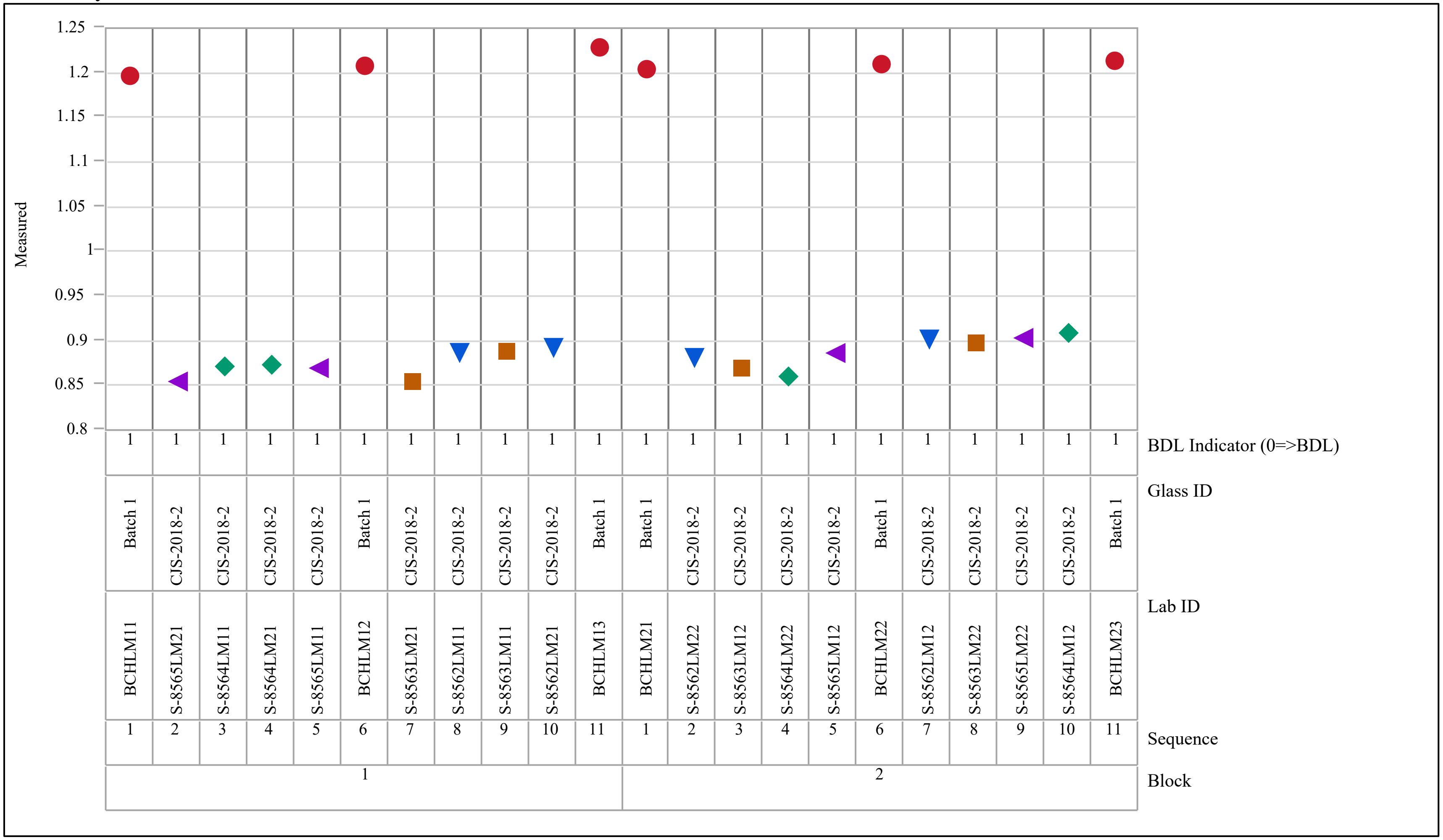


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=Cl (wt%), Prep Method=KH
Variability Chart for Measured

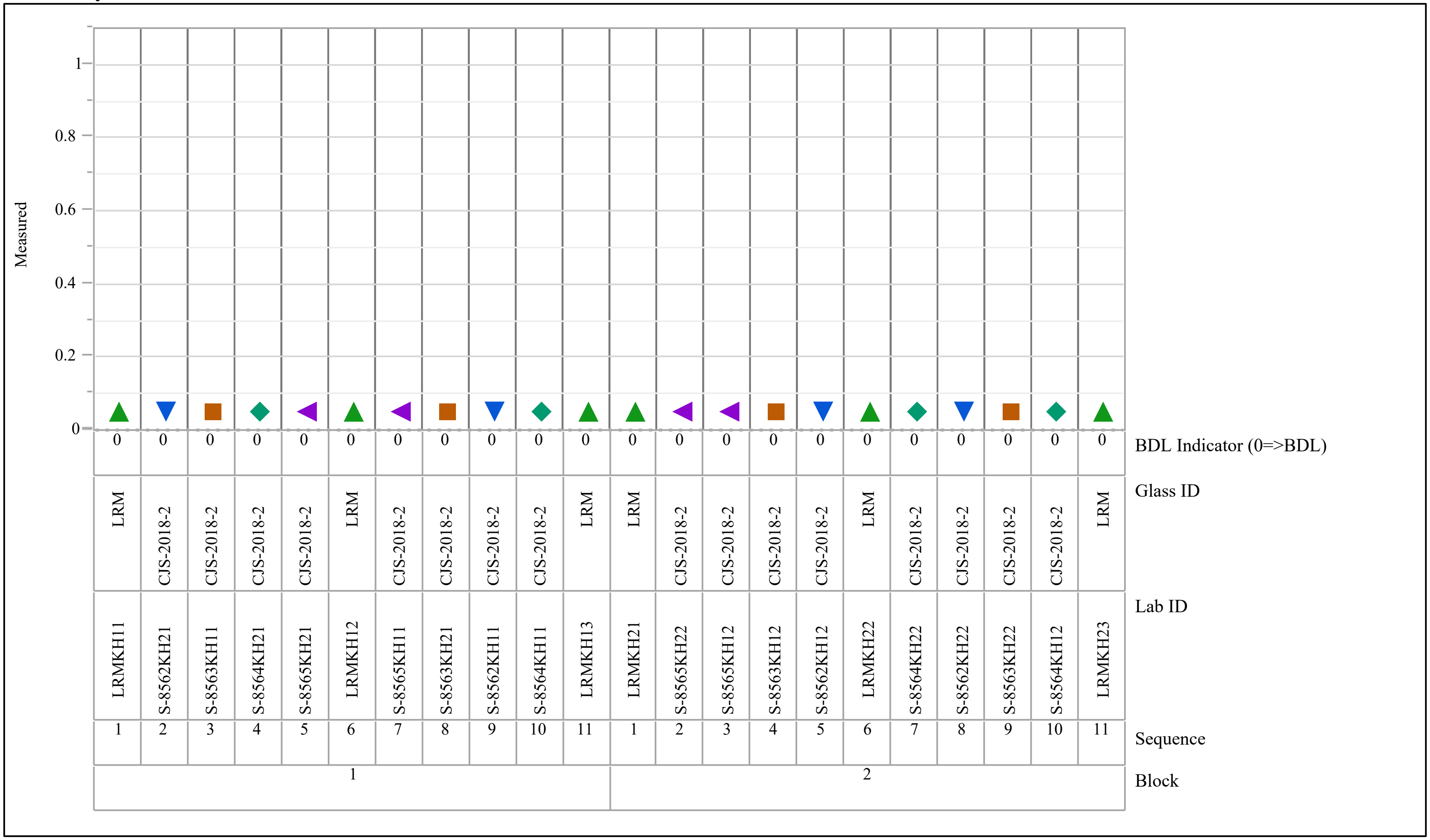


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=Cr2O3 (wt%), Prep Method=LM
Variability Chart for Measured

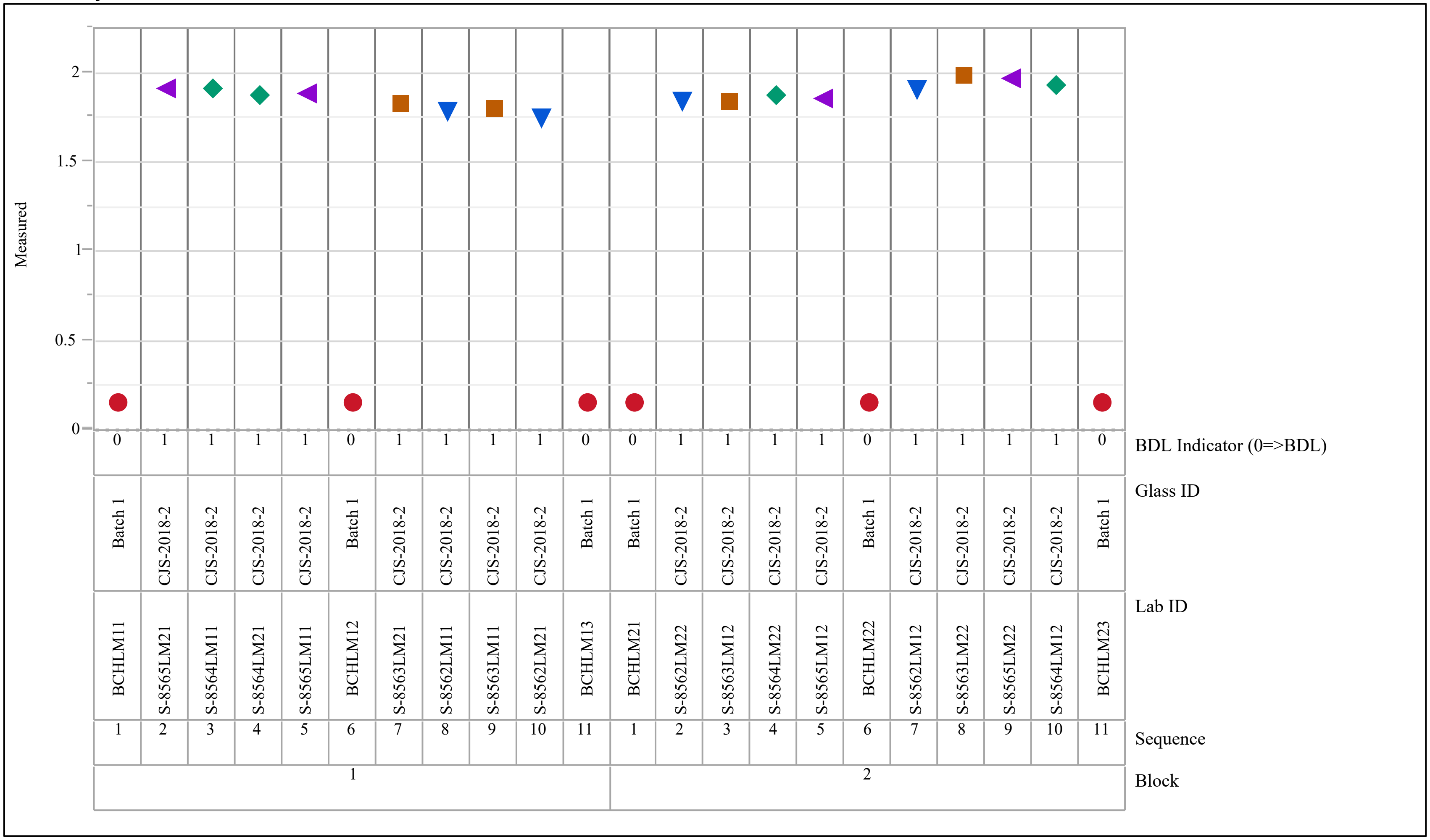


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=F (wt%), Prep Method=KH
Variability Chart for Measured

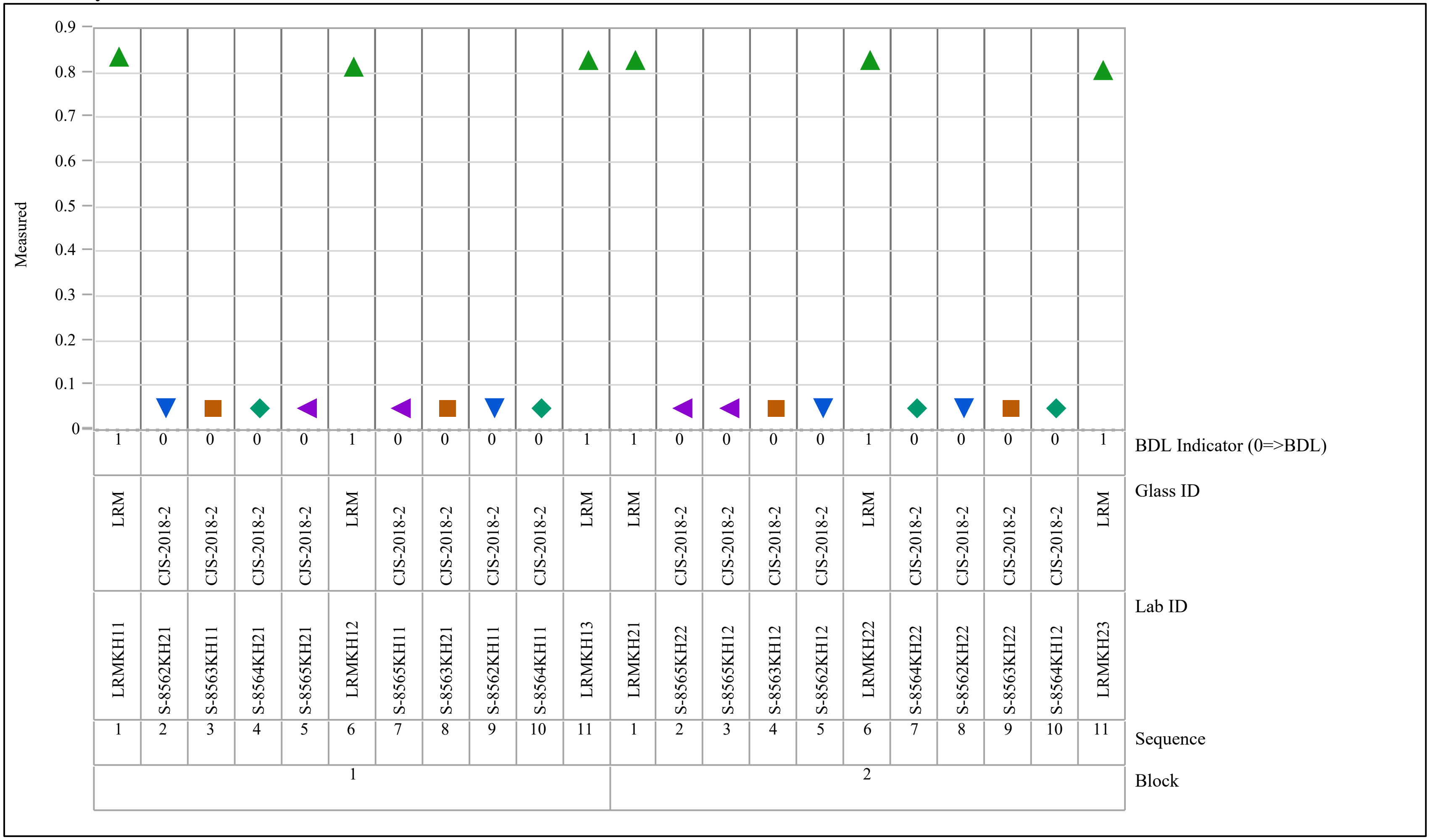


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=Fe2O3 (wt%), Prep Method=PF
Variability Chart for Measured

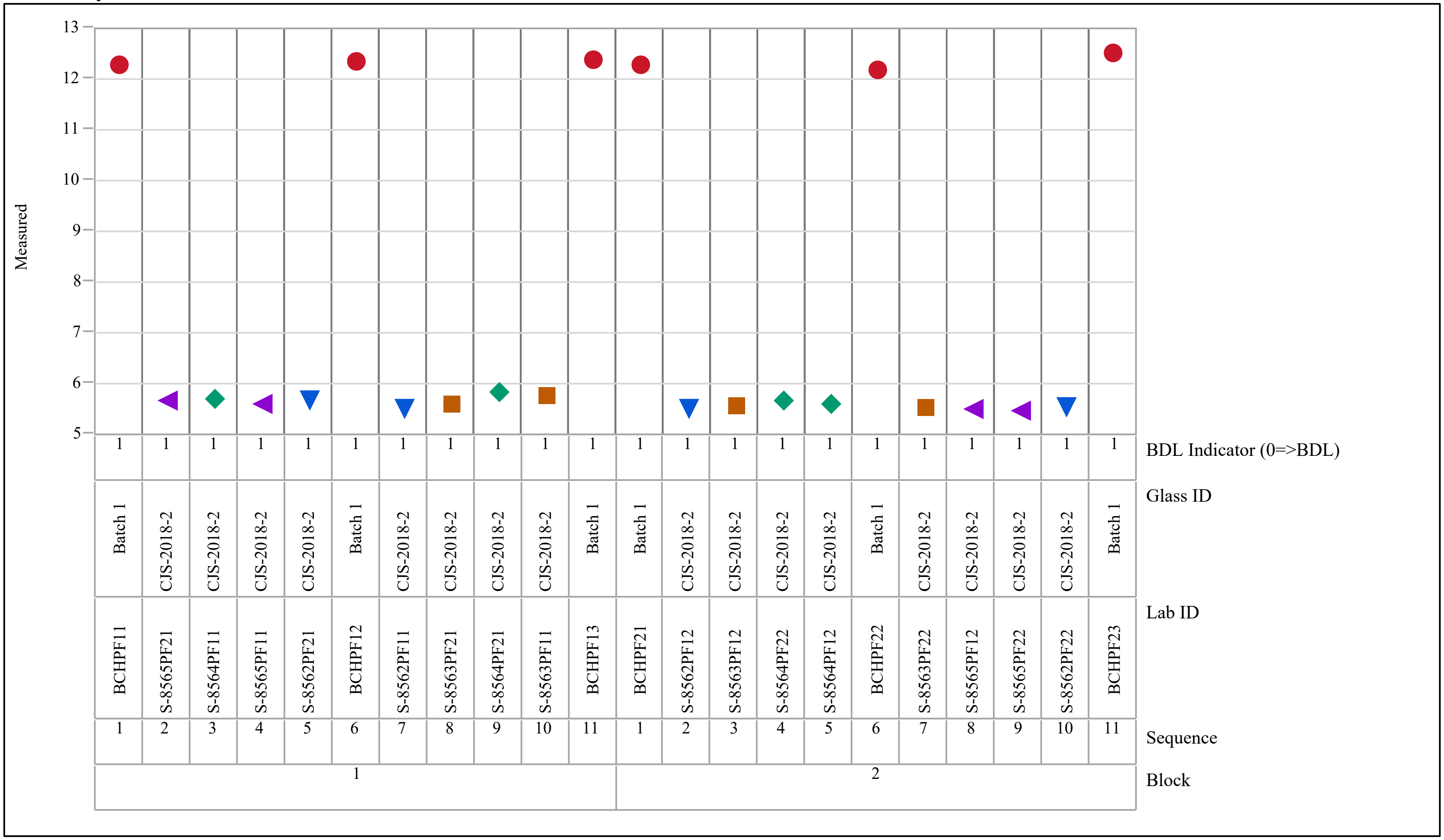


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=K2O (wt%), Prep Method=LM
Variability Chart for Measured

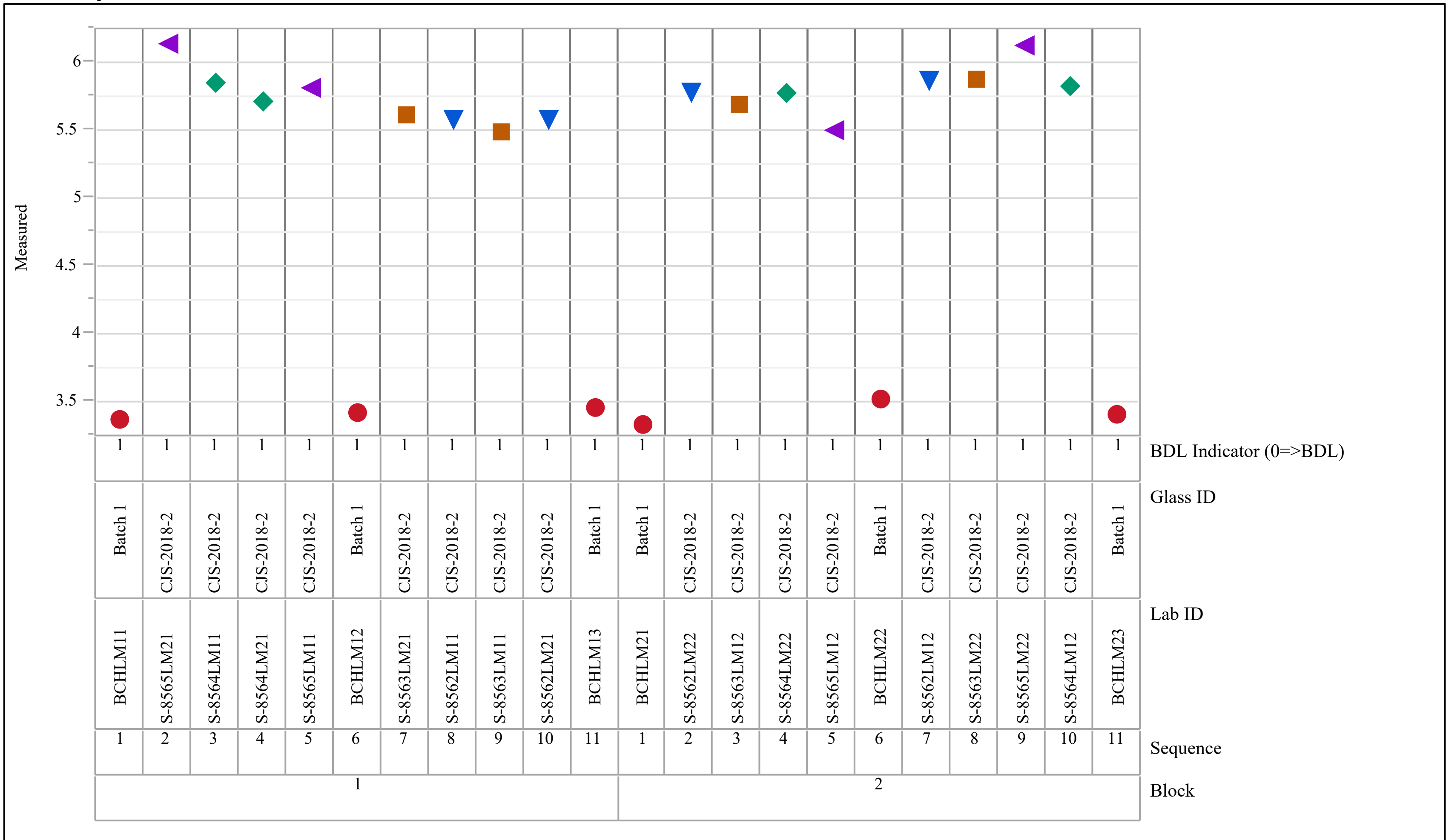


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=Li2O (wt%), Prep Method=PF
Variability Chart for Measured

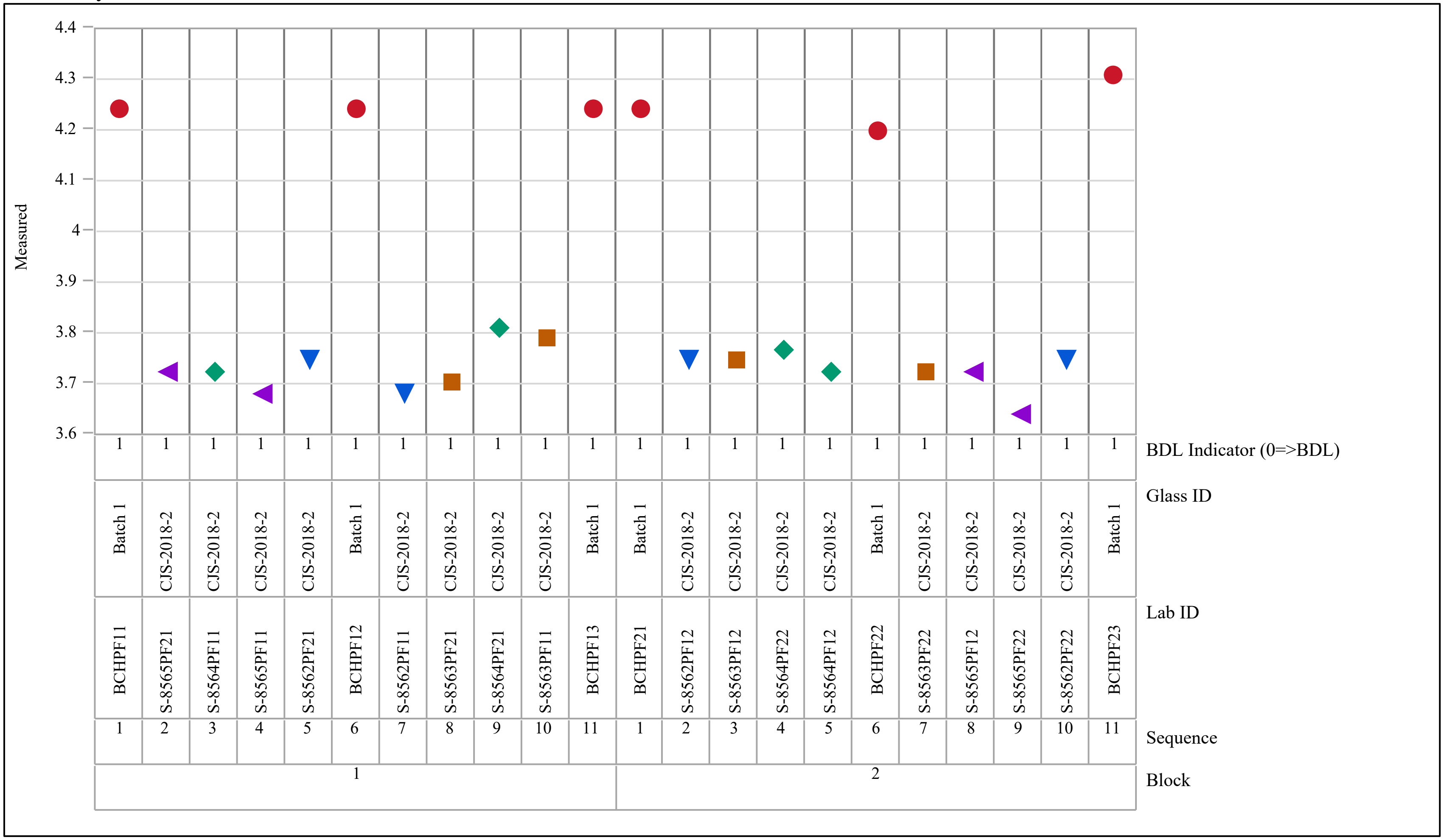


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=MnO (wt%), Prep Method=PF
Variability Chart for Measured

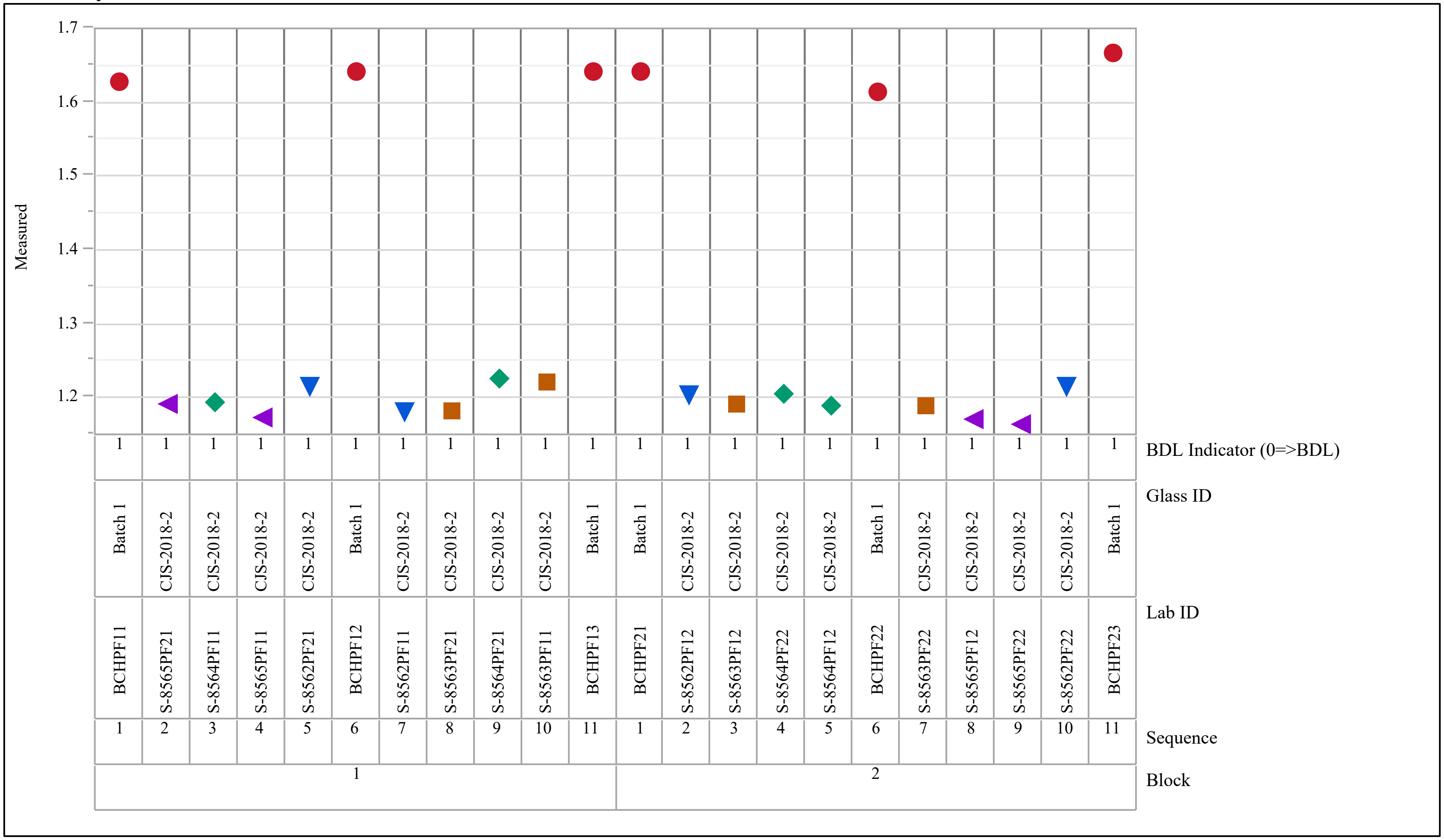


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=Na2O (wt%), Prep Method=LM
Variability Chart for Measured

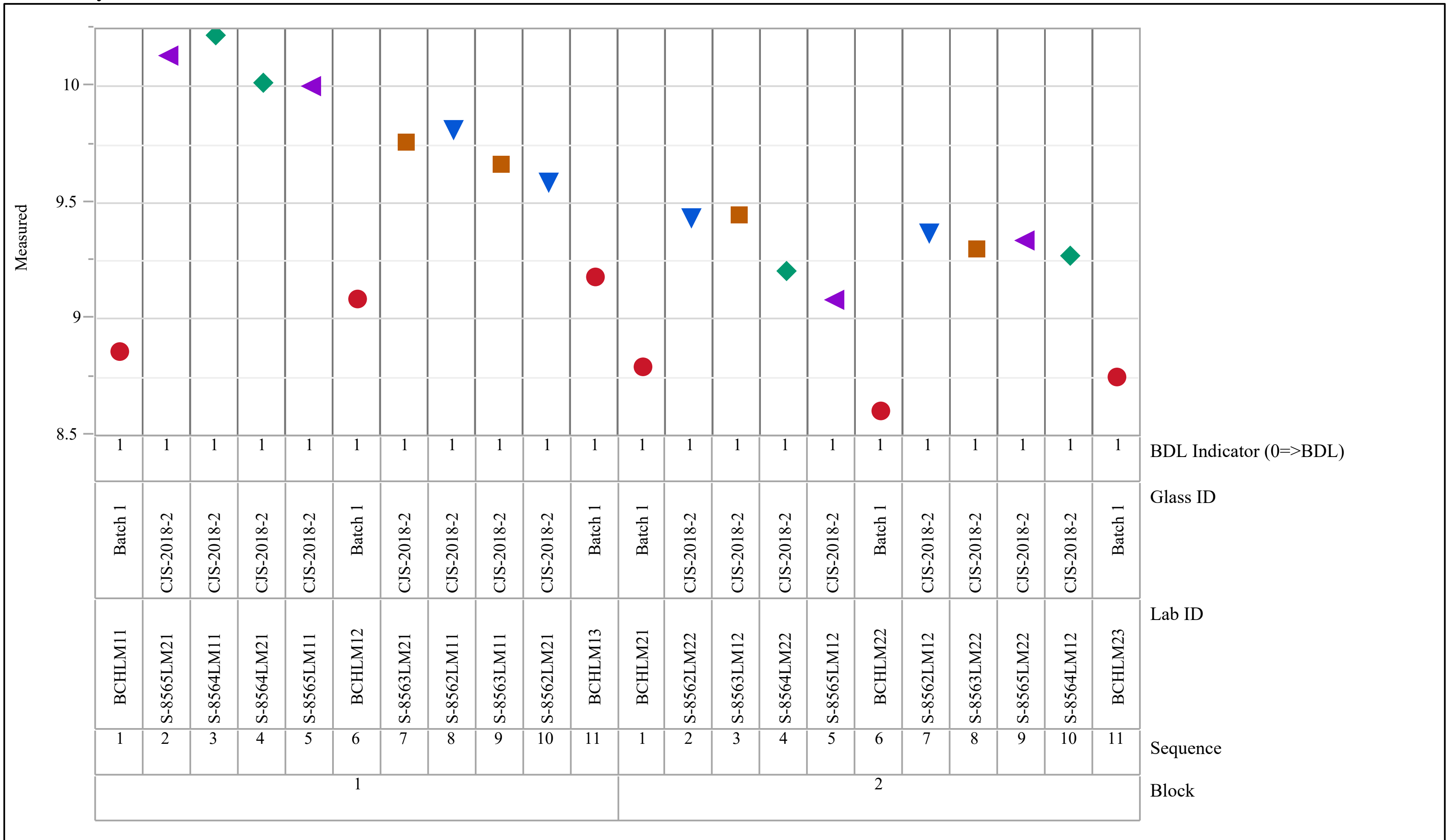


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=NiO (wt%), Prep Method=LM
Variability Chart for Measured

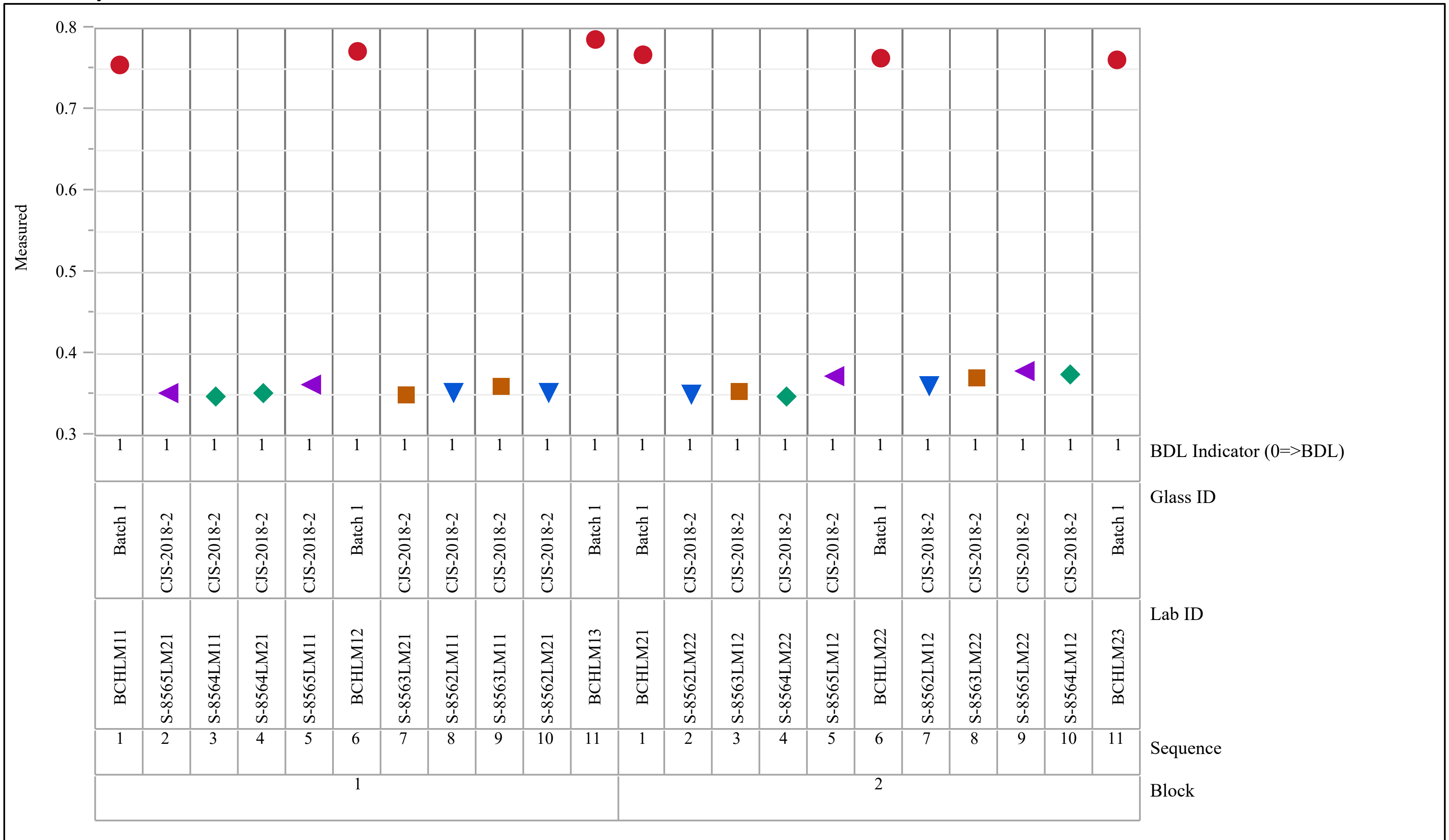


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=P2O5 (wt%), Prep Method=LM
Variability Chart for Measured

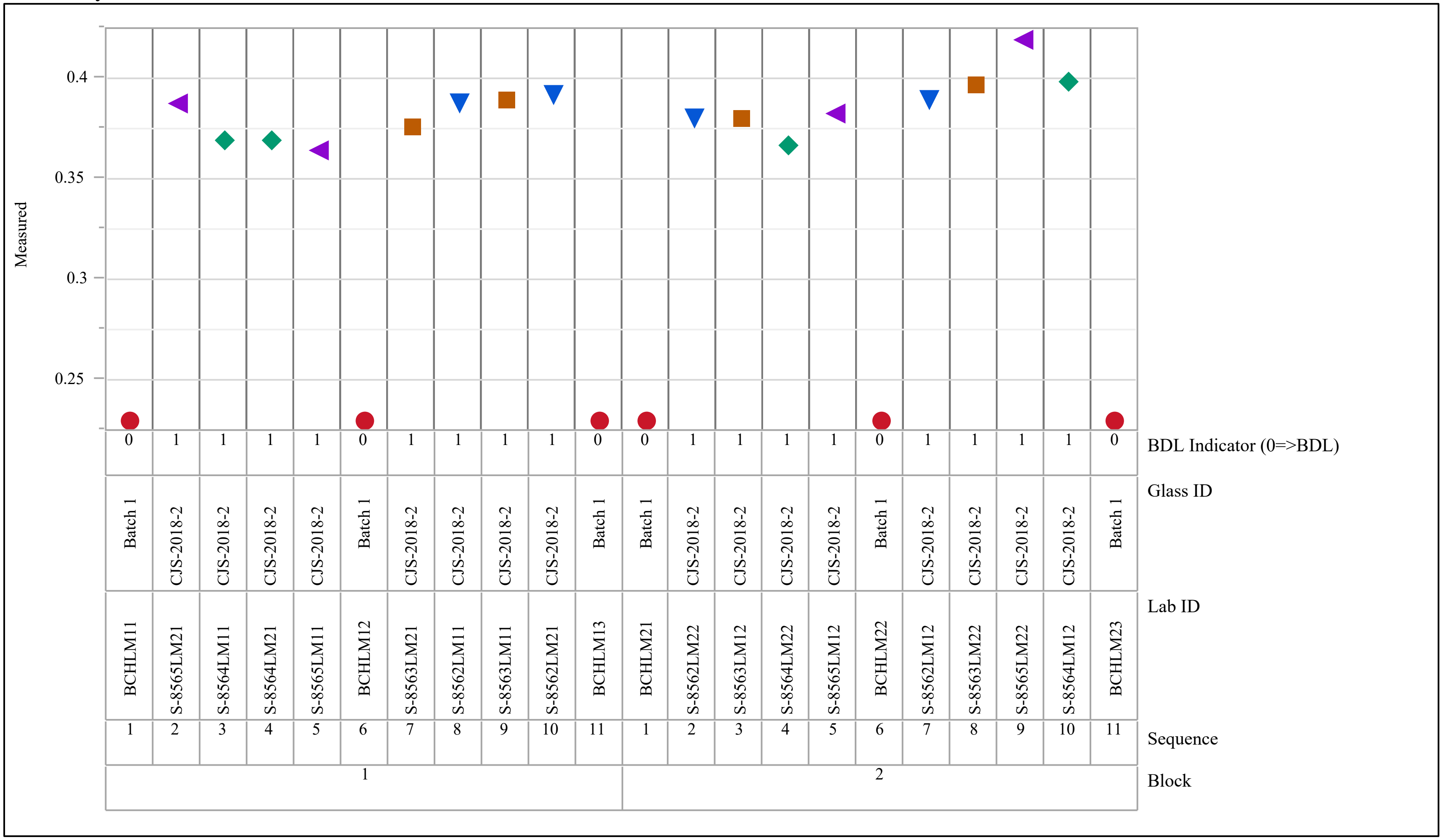


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=PbO (wt%), Prep Method=LM
Variability Chart for Measured

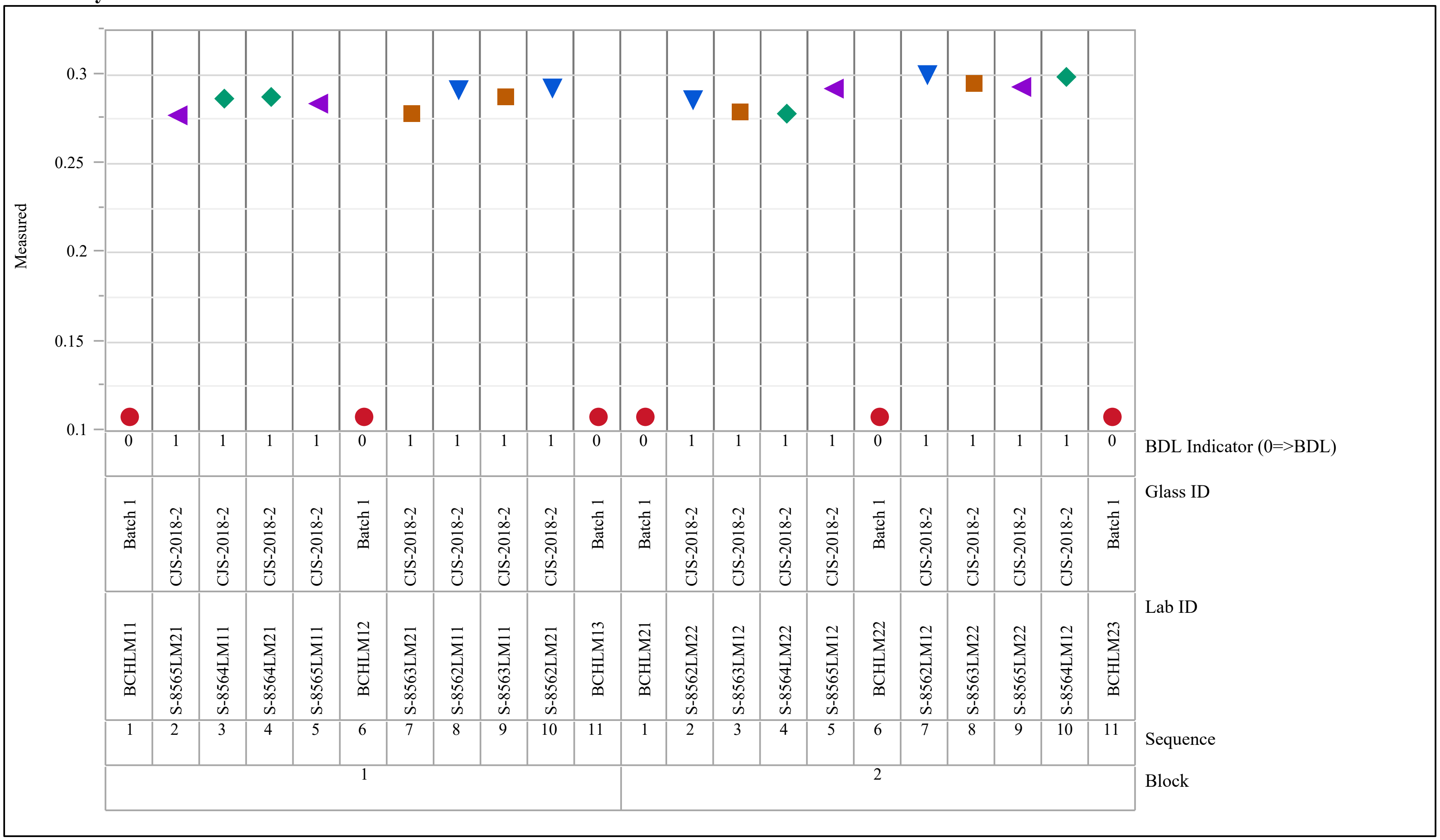


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=SiO2 (wt%), Prep Method=PF
Variability Chart for Measured

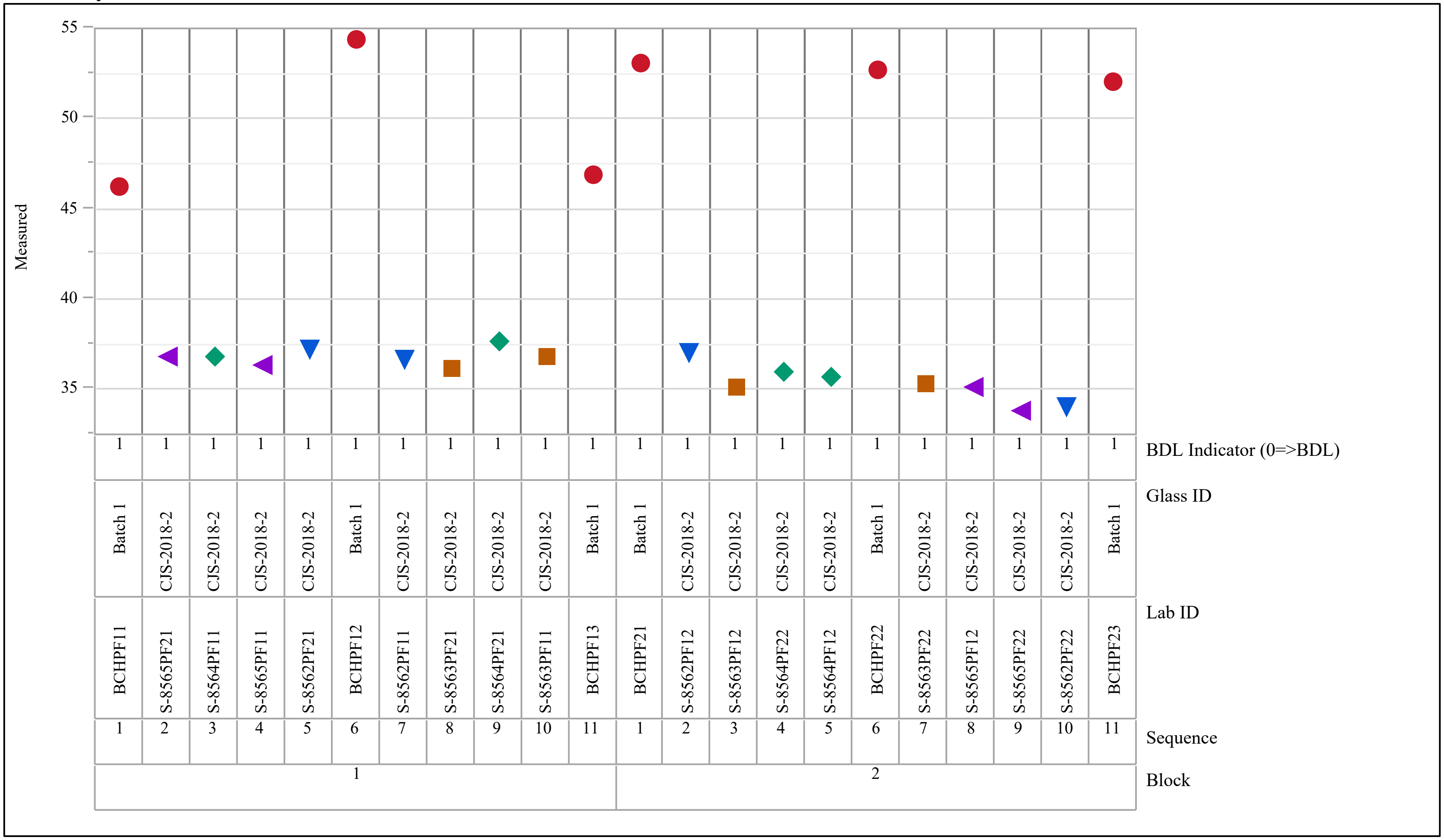


Exhibit A-1. Plots of Oxide Measurements in Analytical Sequence (continued)

Analyte=WO3 (wt%), Prep Method=LM
Variability Chart for Measured

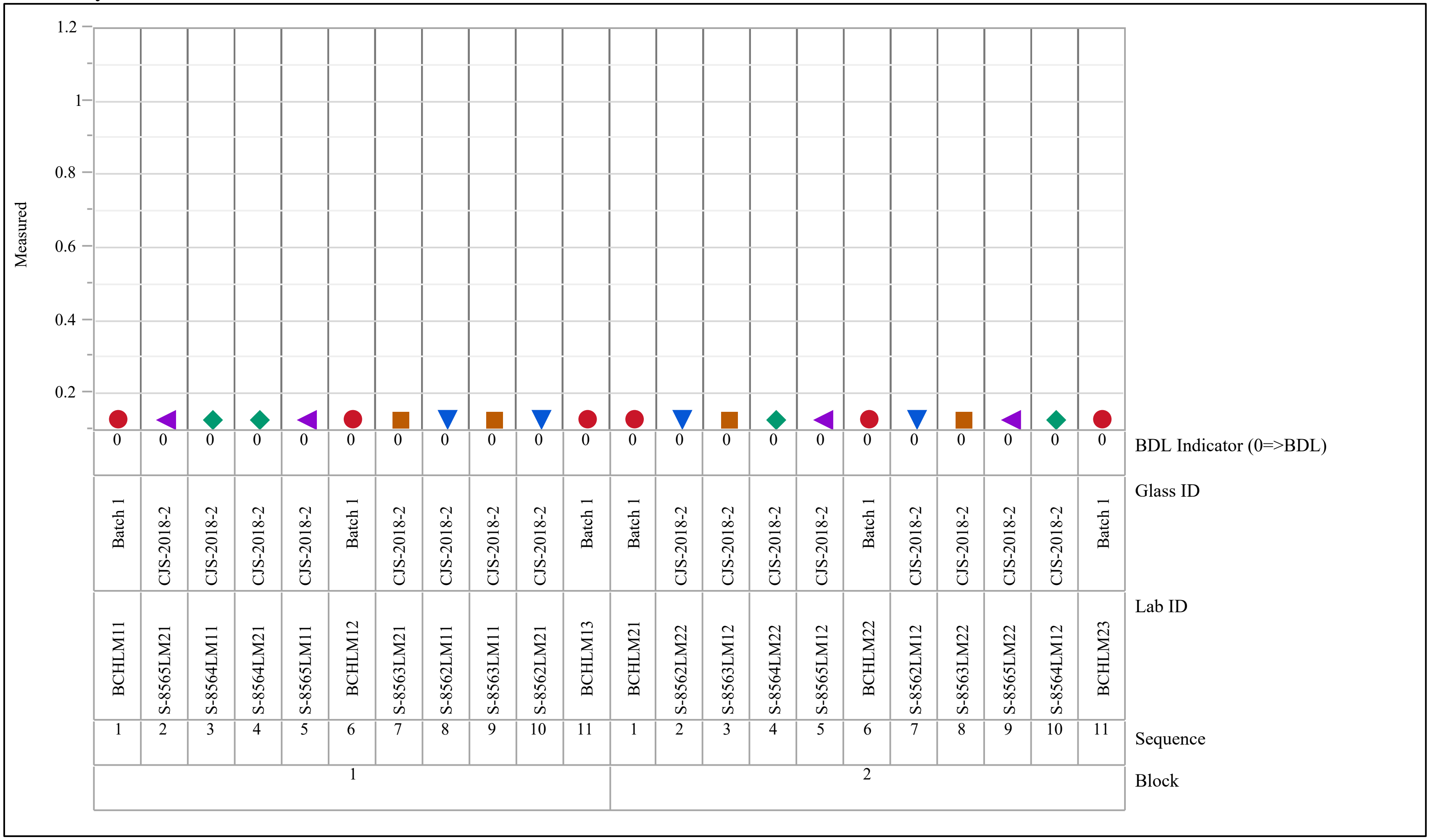
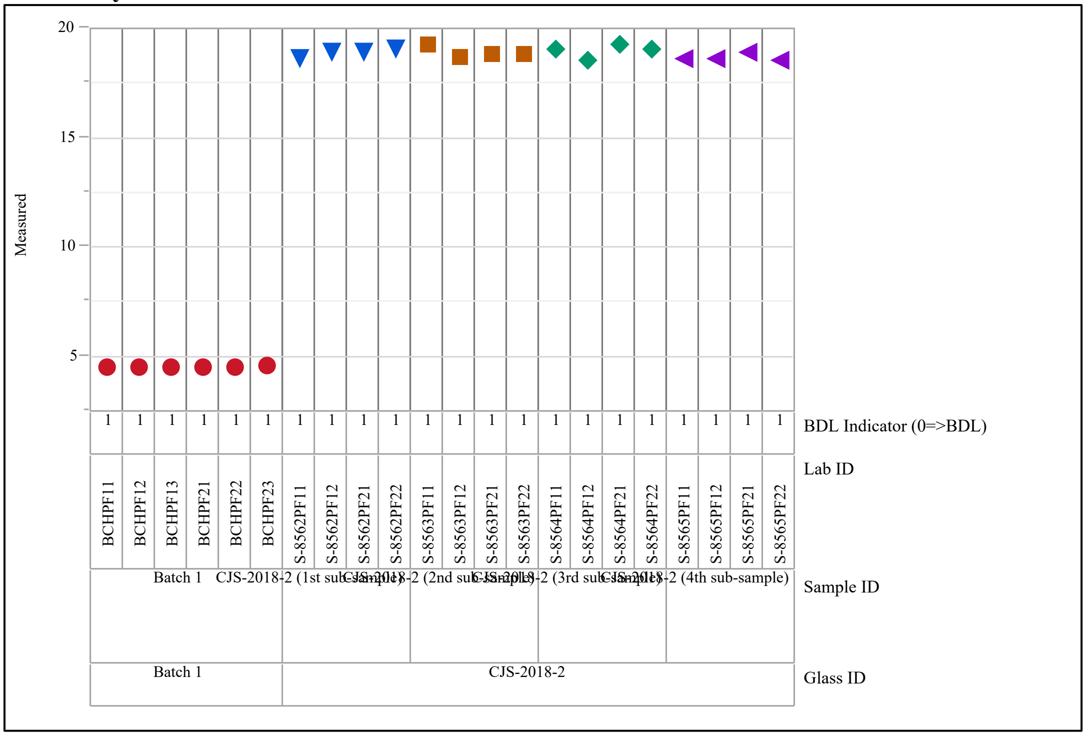


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier

Analyte=Al2O3 (wt%), Prep Method=PF
Variability Chart for Measured



Analyte=B2O3 (wt%), Prep Method=PF
Variability Chart for Measured

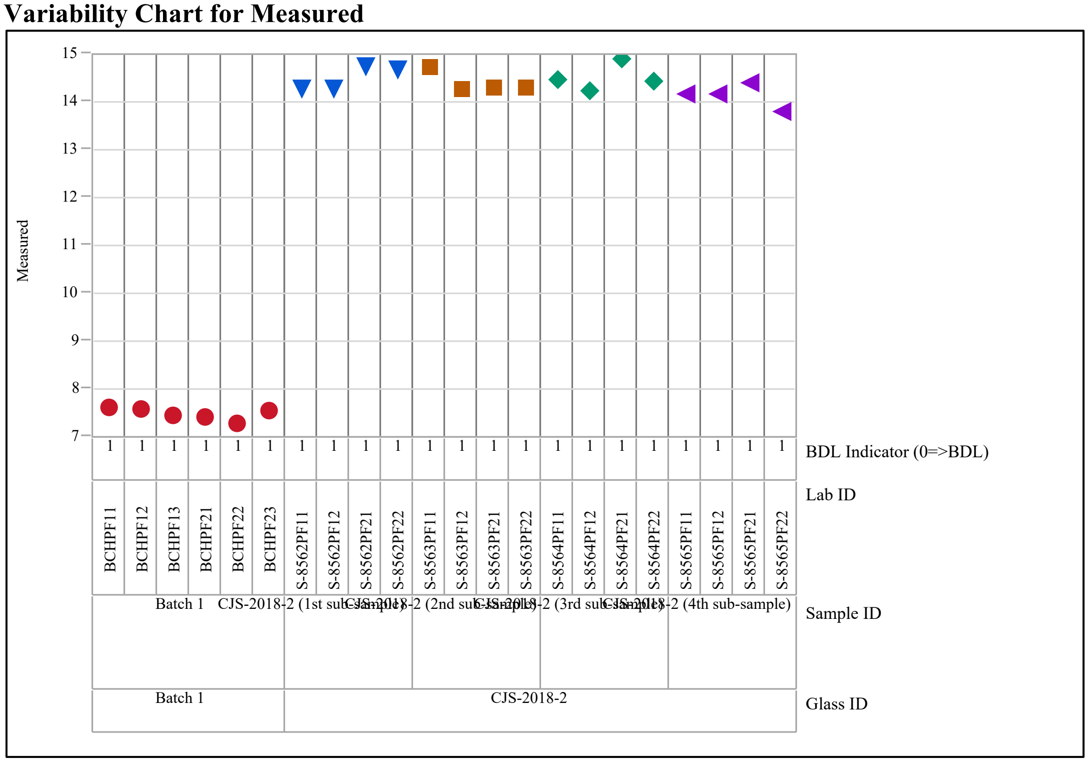
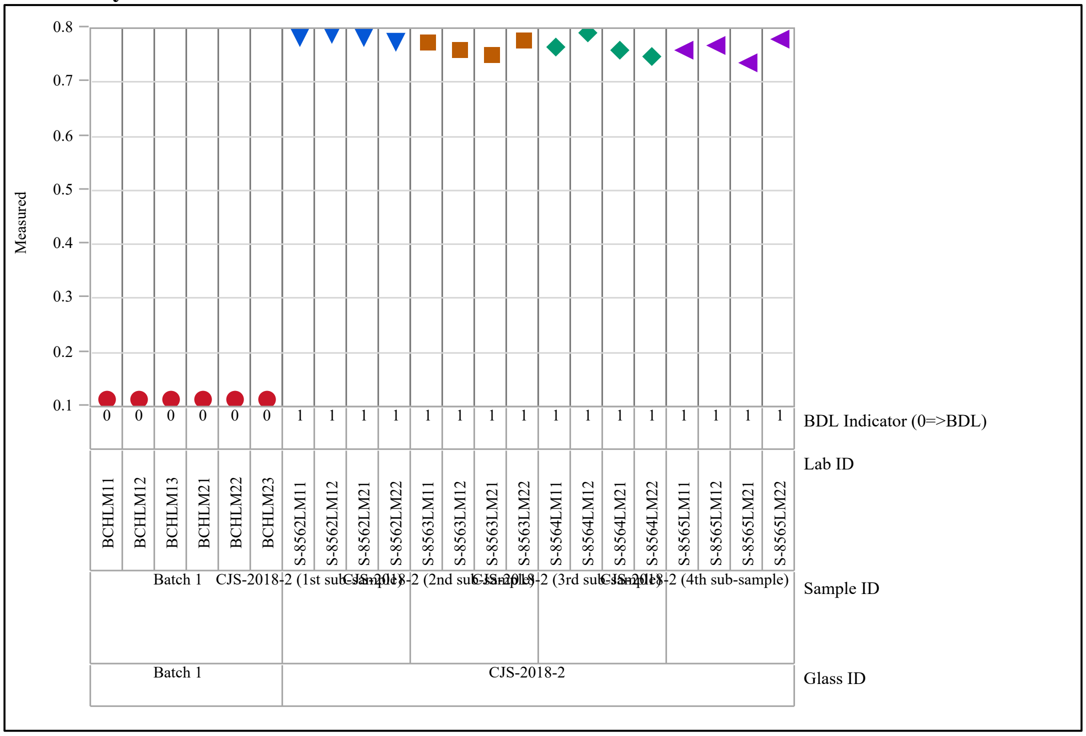


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier (continued)

Analyte=Bi2O3 (wt%), Prep Method=LM
Variability Chart for Measured



Analyte=CaO (wt%), Prep Method=LM
Variability Chart for Measured

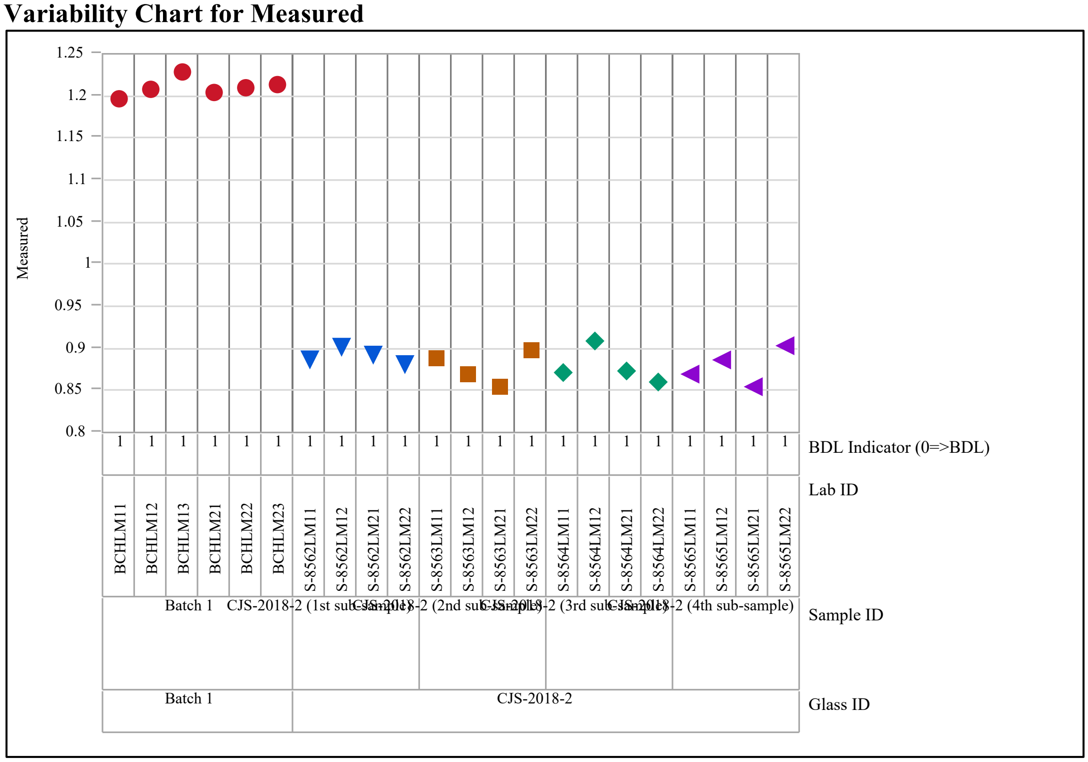
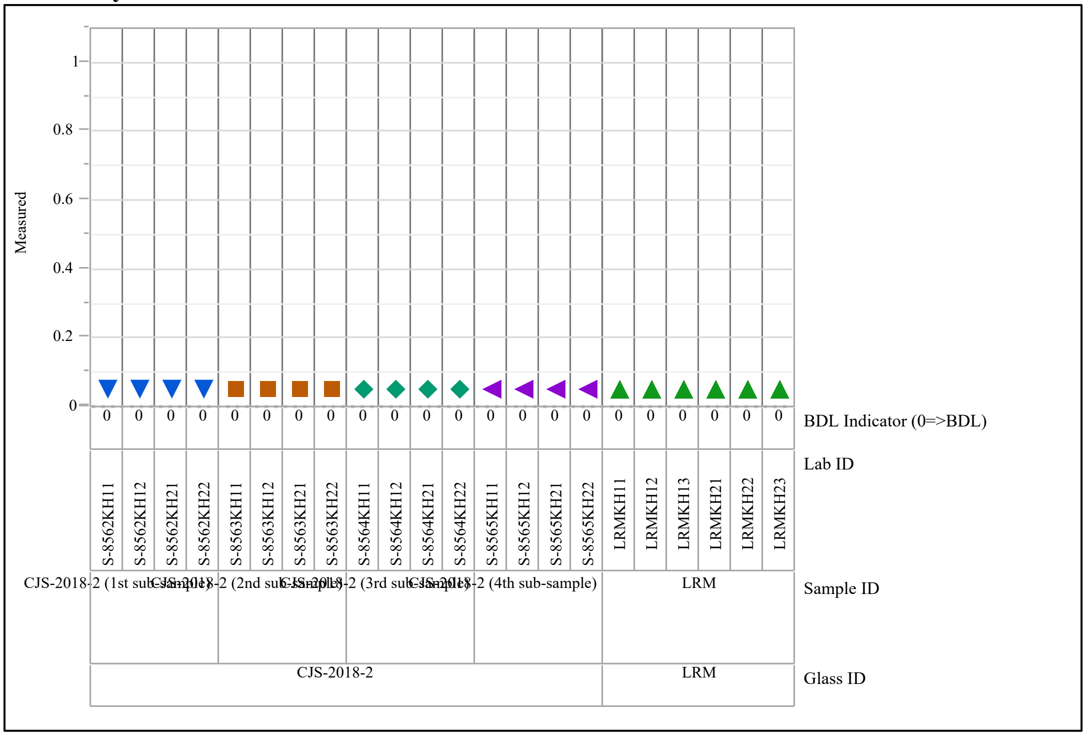


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier (continued)

Analyte=Cl (wt%), Prep Method=KH
Variability Chart for Measured



Analyte=Cr2O3 (wt%), Prep Method=LM
Variability Chart for Measured

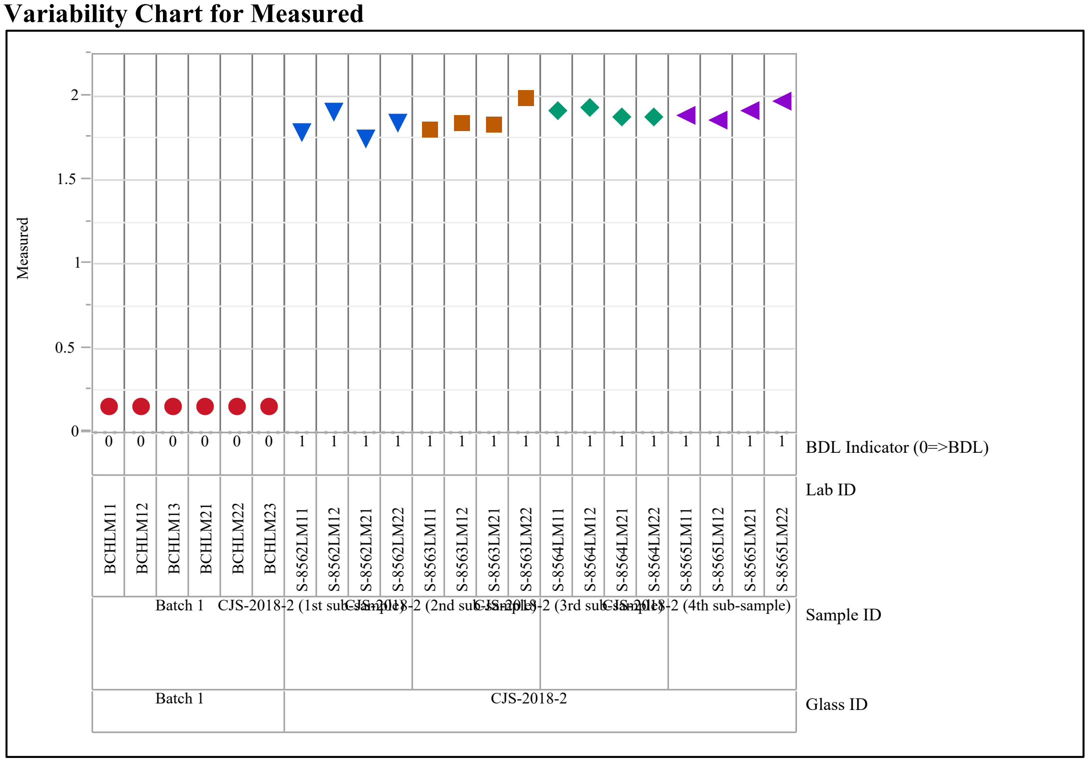
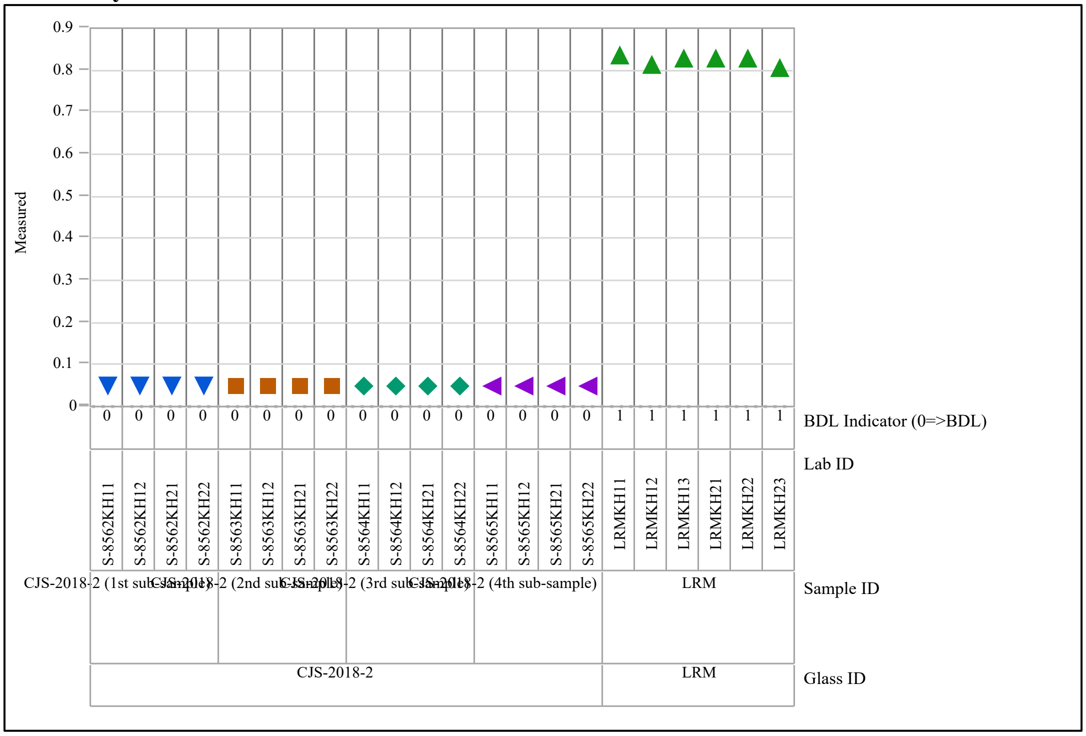


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier (continued)

Analyte=F (wt%), Prep Method=KH
Variability Chart for Measured



Analyte=Fe2O3 (wt%), Prep Method=PF
Variability Chart for Measured

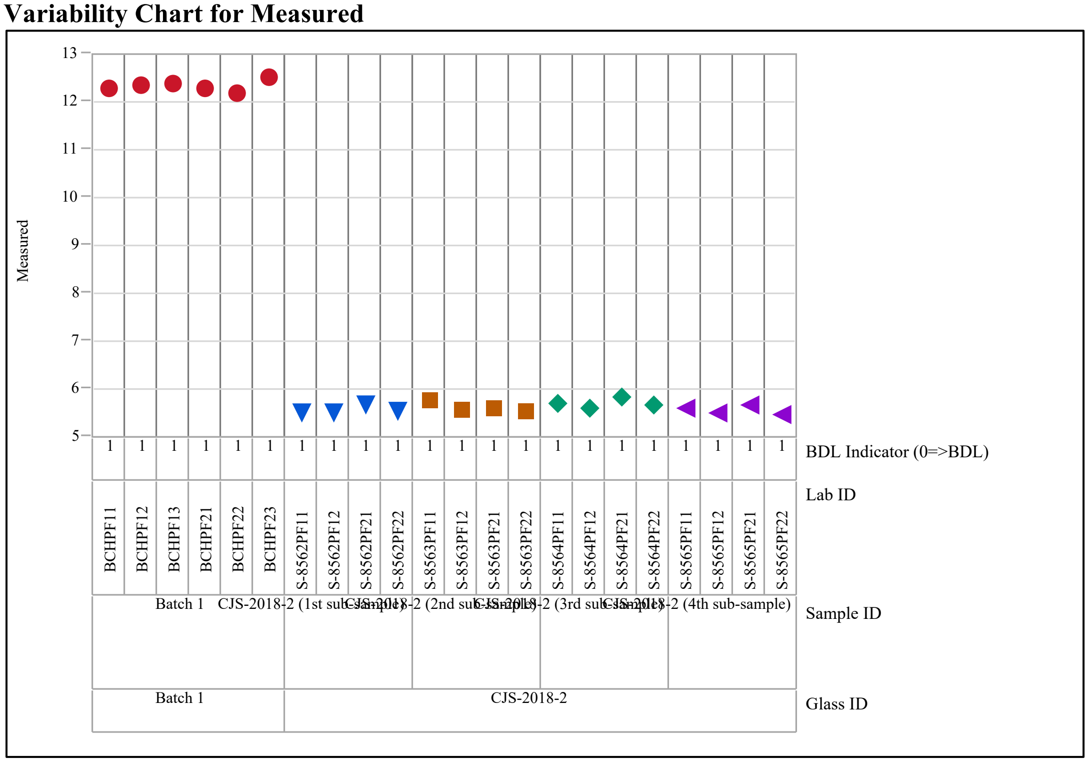
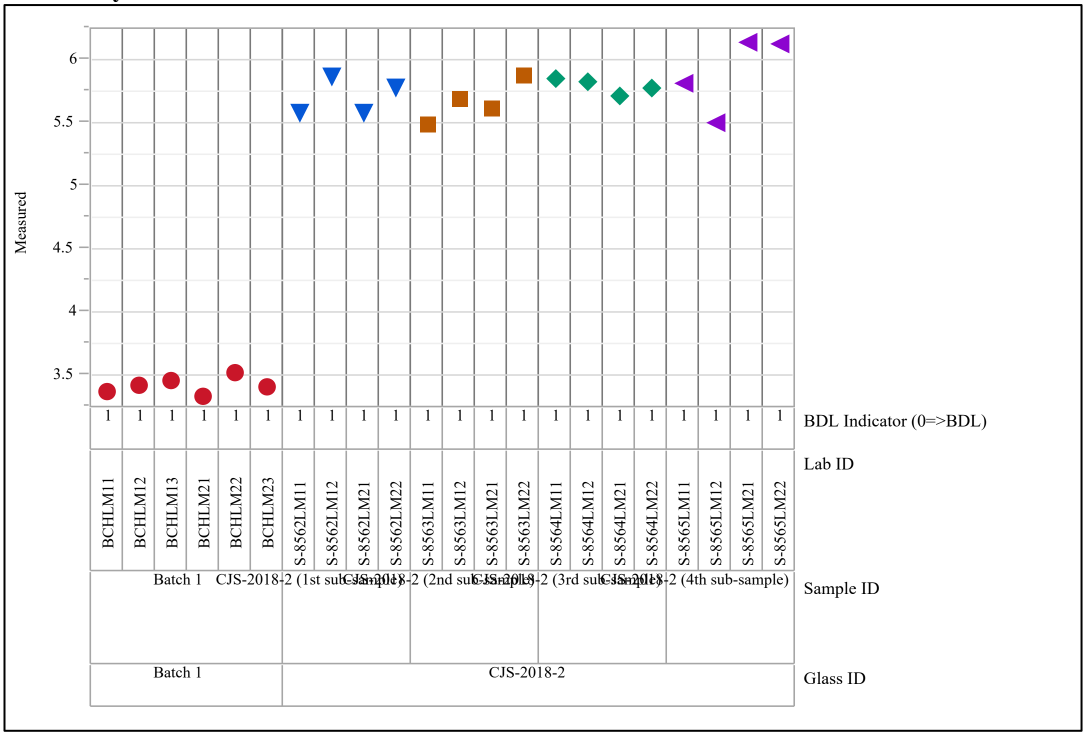


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier (continued)

Analyte=K2O (wt%), Prep Method=LM
Variability Chart for Measured



Analyte=Li2O (wt%), Prep Method=PF
Variability Chart for Measured

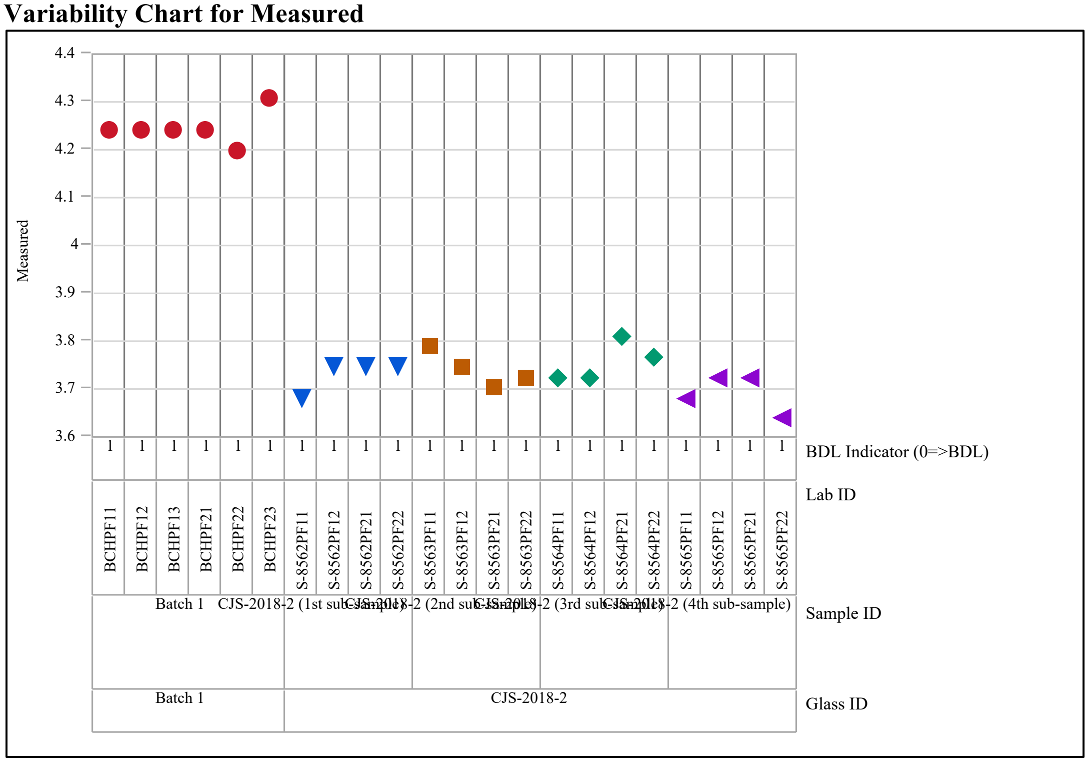
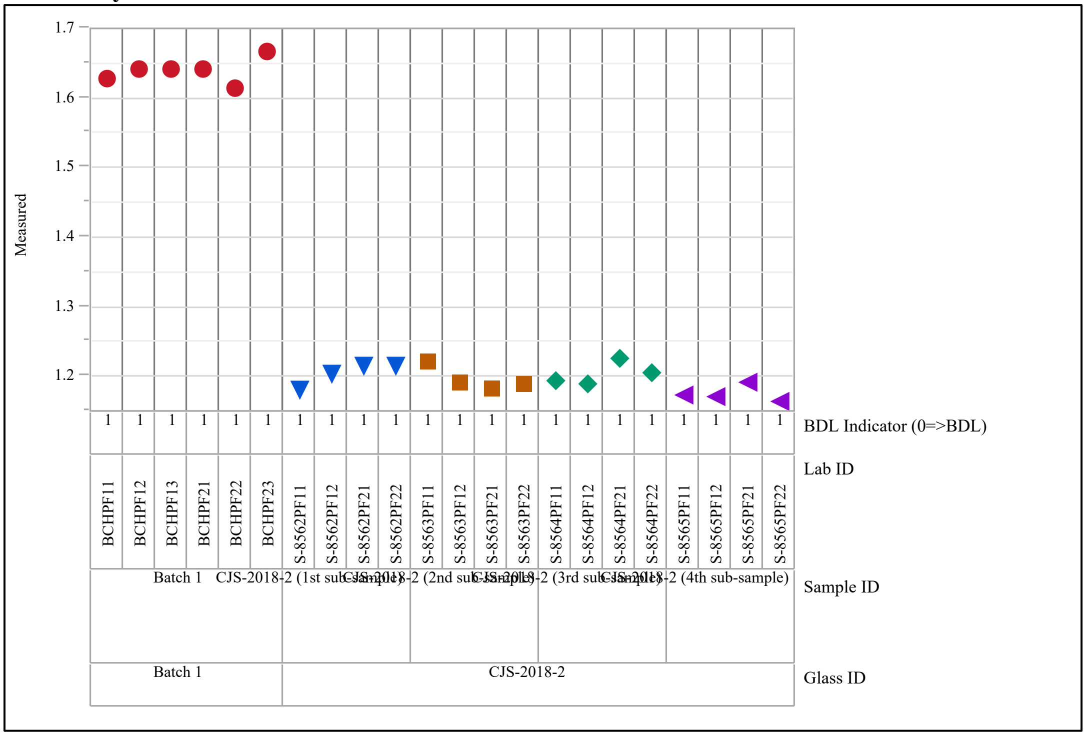


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier (continued)

Analyte=MnO (wt%), Prep Method=PF
Variability Chart for Measured



Analyte=Na2O (wt%), Prep Method=LM
Variability Chart for Measured

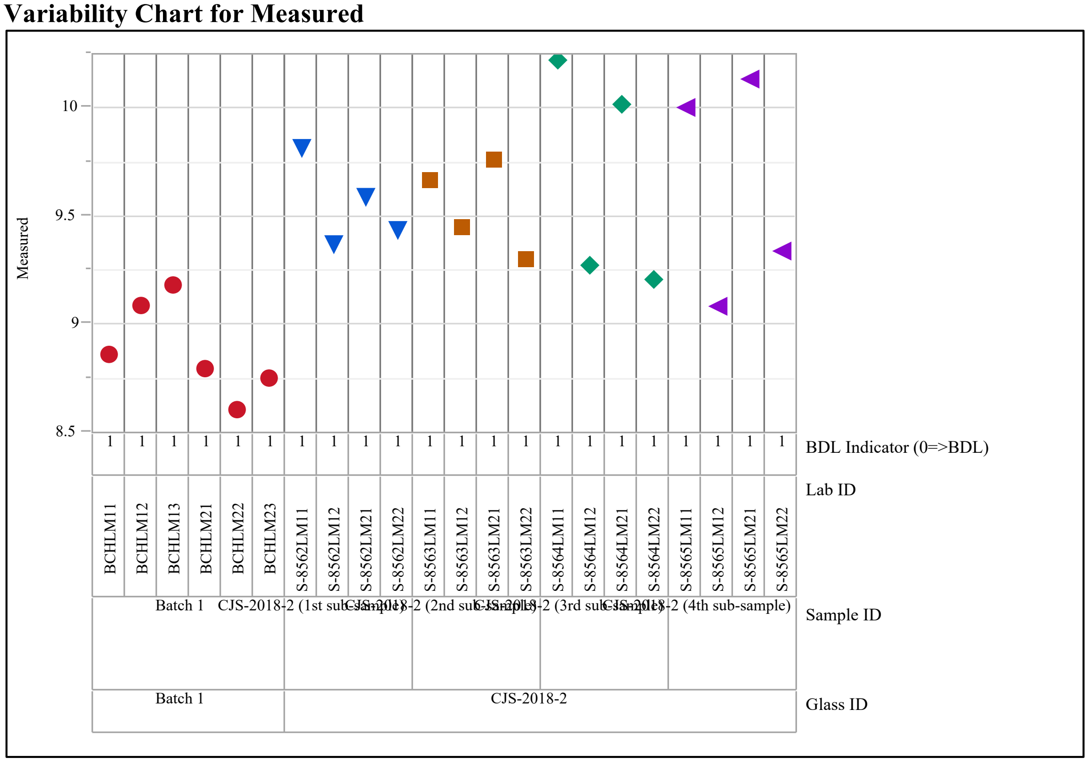
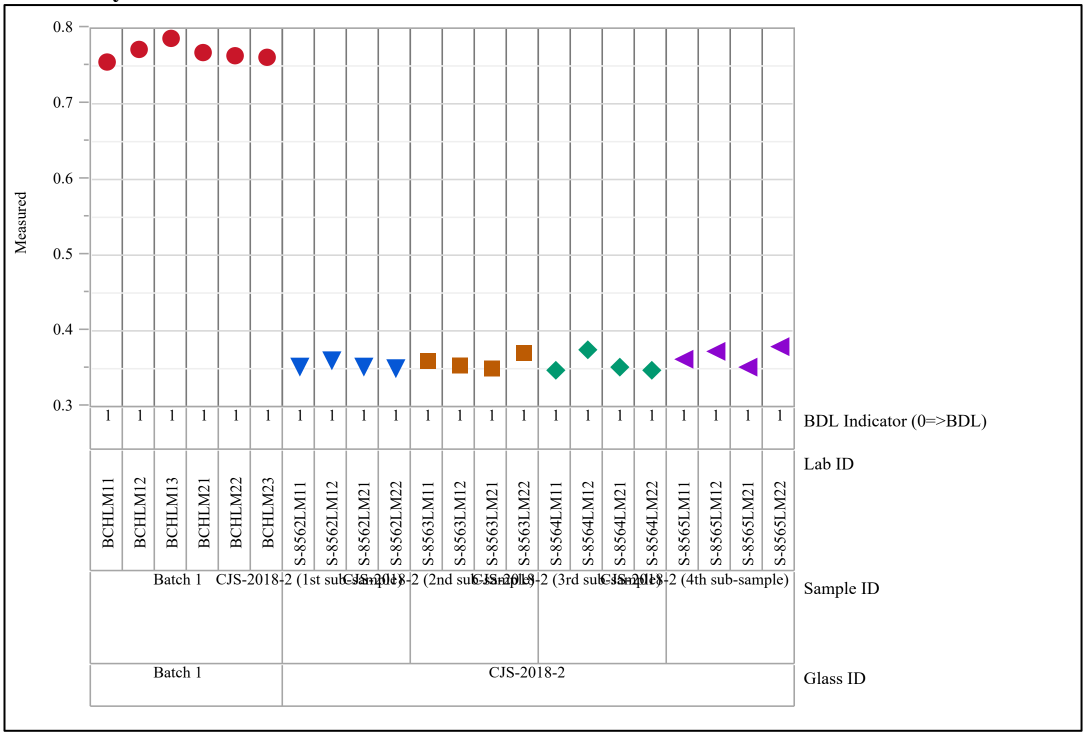


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier (continued)

Analyte=NiO (wt%), Prep Method=LM
Variability Chart for Measured



Analyte=P2O5 (wt%), Prep Method=LM
Variability Chart for Measured

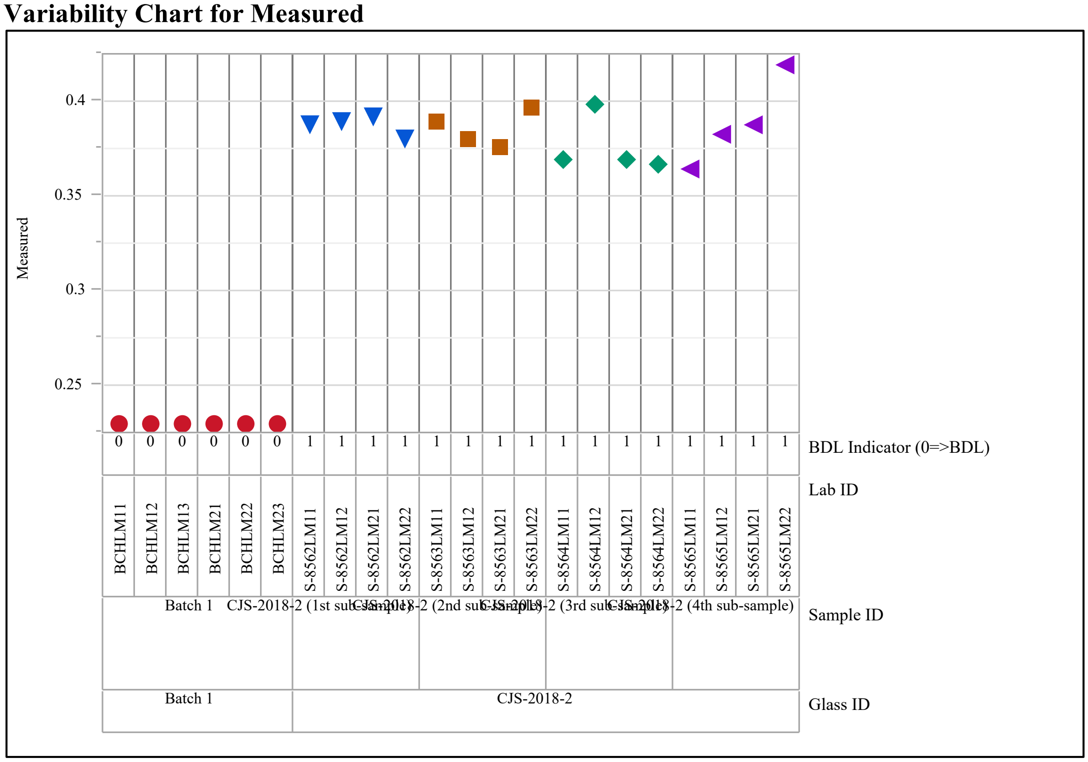
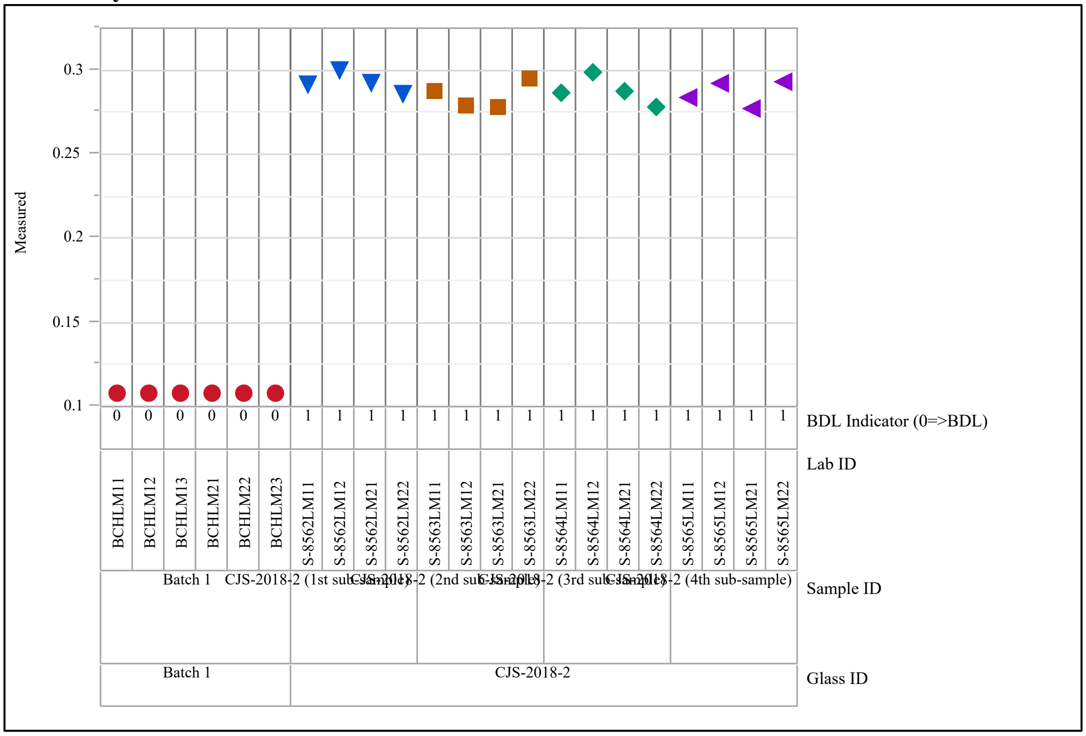


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier (continued)

Analyte=PbO (wt%), Prep Method=LM
Variability Chart for Measured



Analyte=SiO2 (wt%), Prep Method=PF
Variability Chart for Measured

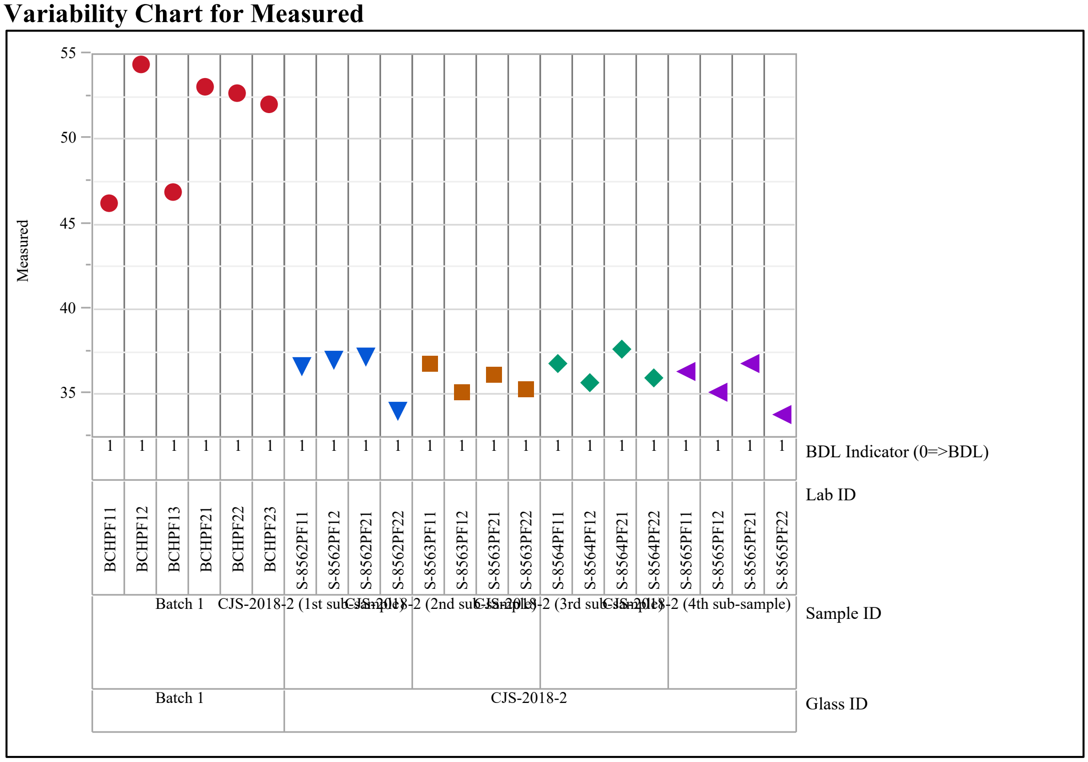


Exhibit A-2. Plots of Oxide Measurements by Sample Identifier (continued)

Analyte=WO3 (wt%), Prep Method=LM
Variability Chart for Measured

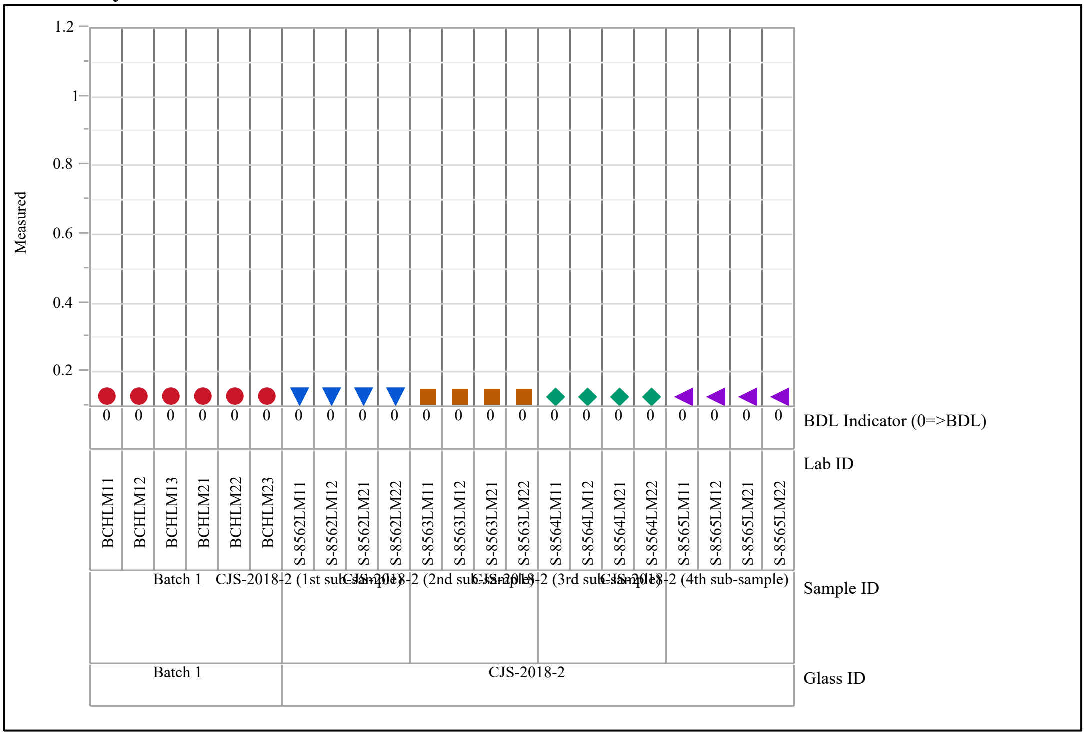
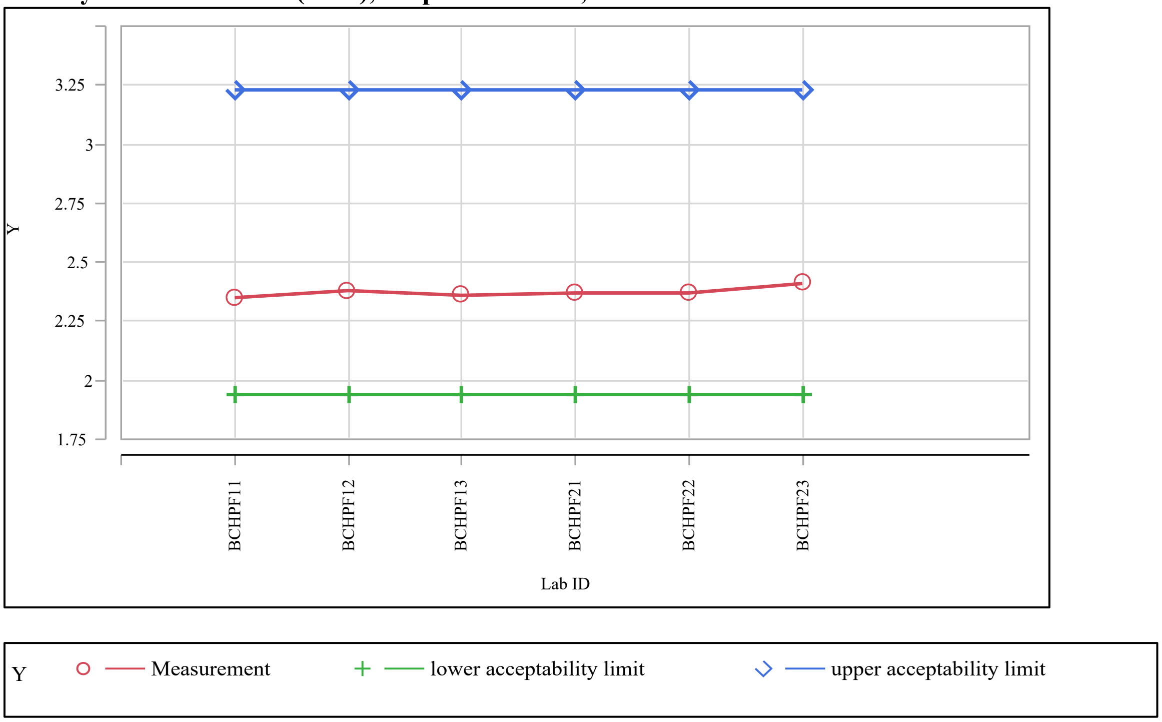


Exhibit A-3. Acceptability Evaluation for Measurements of the Standard Glasses

Overlay Plot Element=Al (wt%), Prep Method=PF, ID=Batch 1



Overlay Plot Element=B (wt%), Prep Method=PF, ID=Batch 1

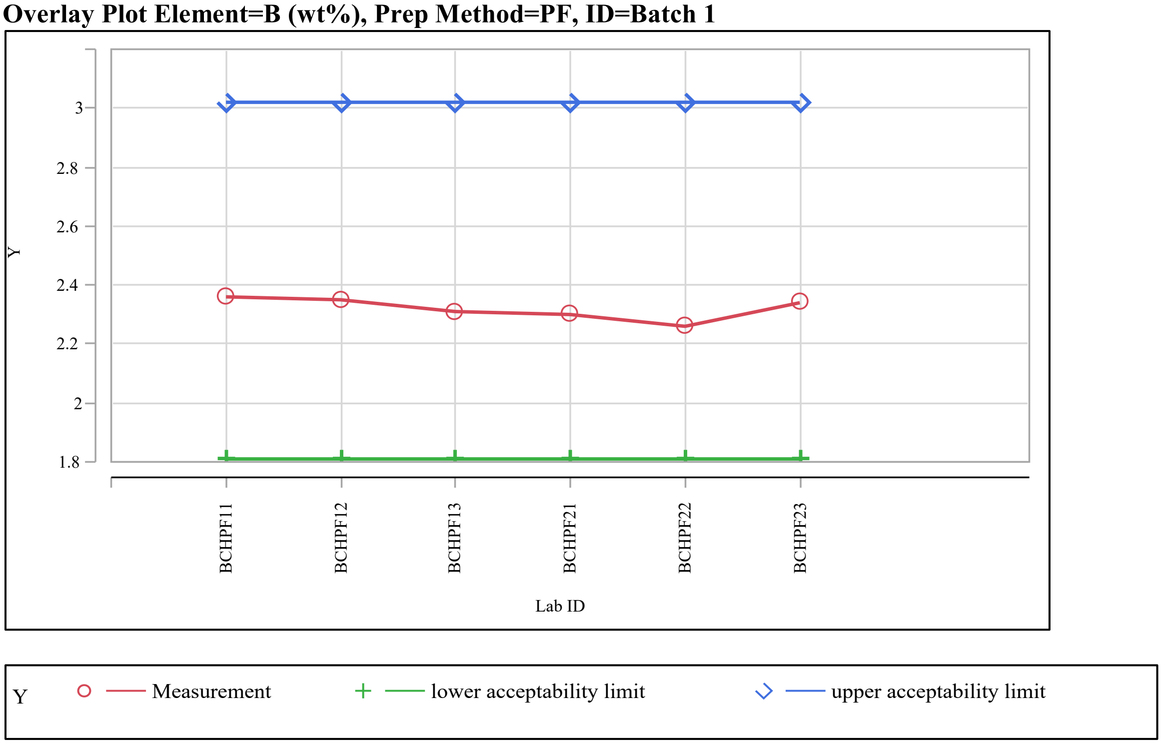
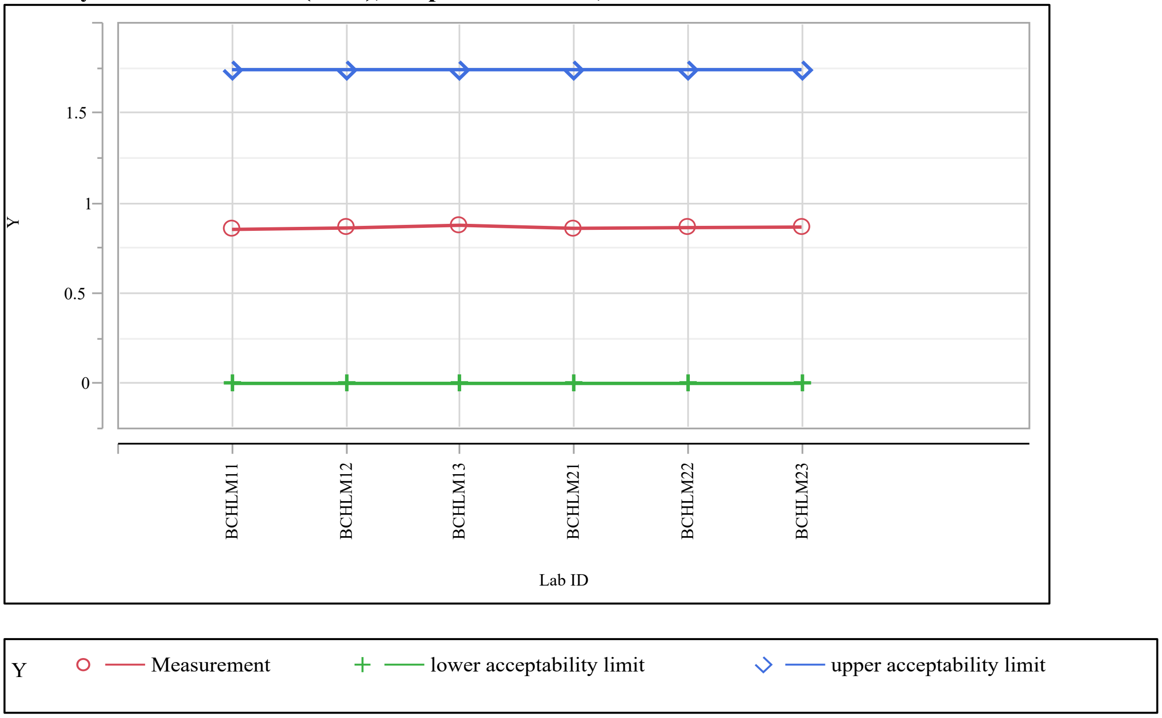


Exhibit A-3. Acceptability Evaluation for Measurements of the Standard Glass (continued)

Overlay Plot Element=Ca (wt%), Prep Method=LM, ID=Batch 1



Overlay Plot Element=F (wt%), Prep Method=KH, ID=LRM

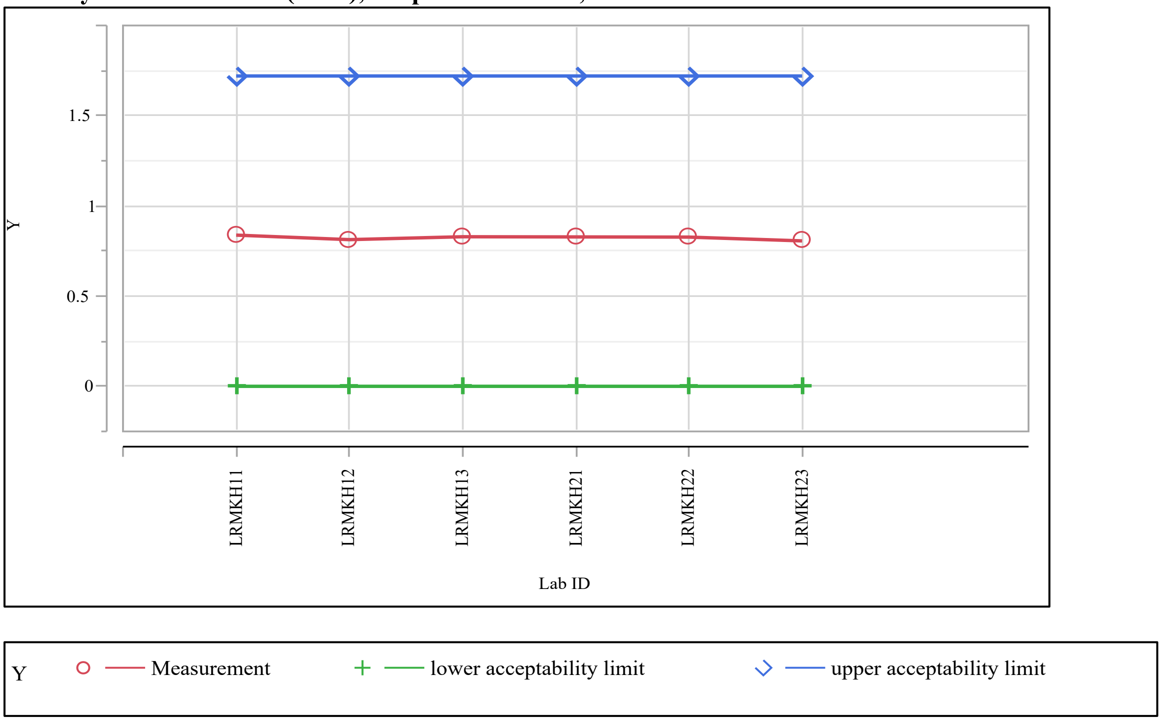
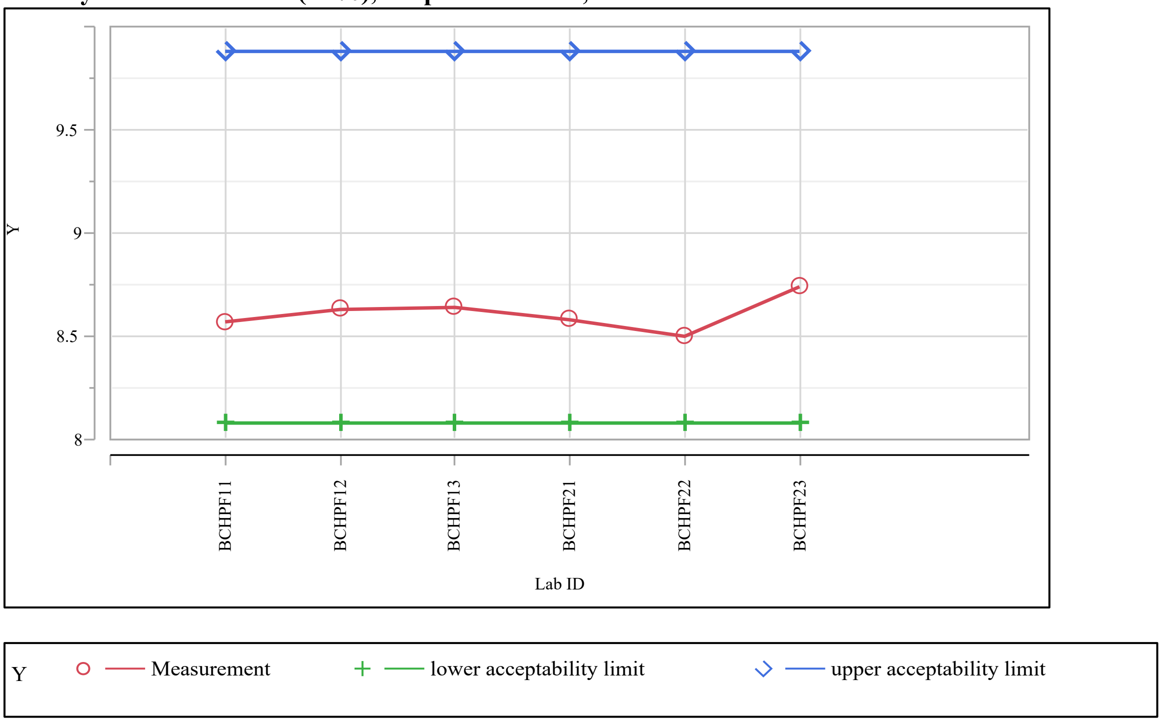


Exhibit A-3. Acceptability Evaluation for Measurements of the Standard Glass (continued)

Overlay Plot Element=Fe (wt%), Prep Method=PF, ID=Batch 1



Overlay Plot Element=K (wt%), Prep Method=LM, ID=Batch 1

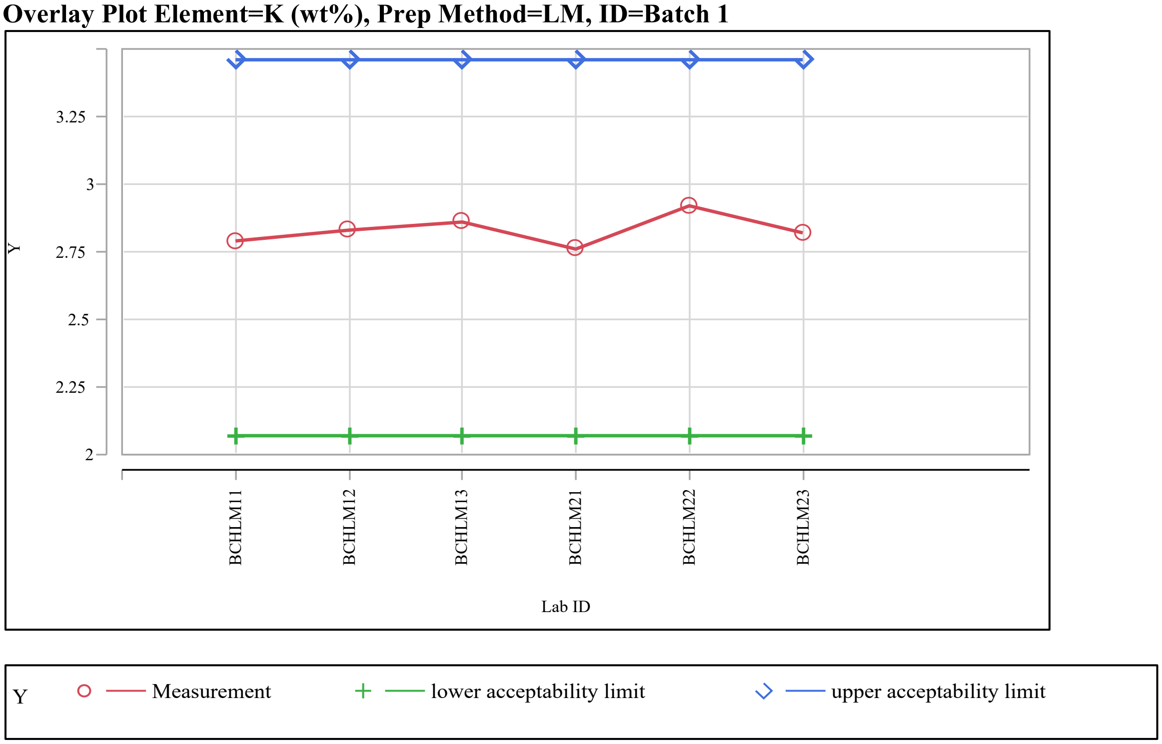
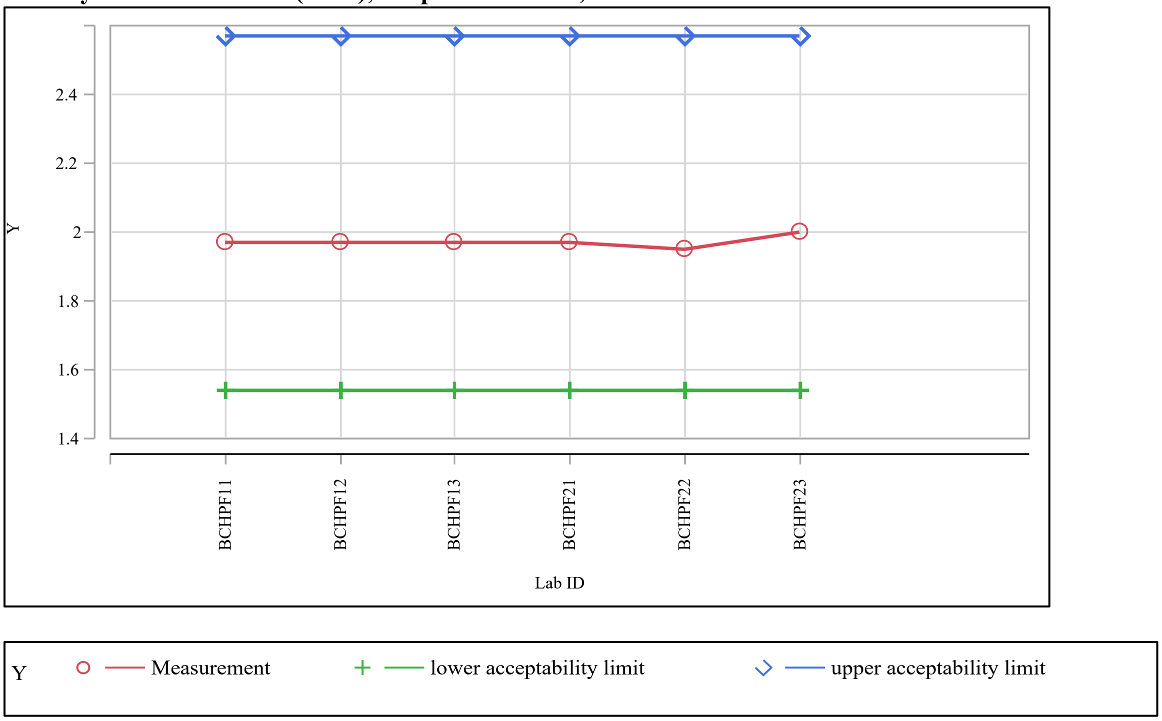


Exhibit A-3. Acceptability Evaluation for Measurements of the Standard Glass (continued)

Overlay Plot Element=Li (wt%), Prep Method=PF, ID=Batch 1



Overlay Plot Element=Mn (wt%), Prep Method=PF, ID=Batch 1

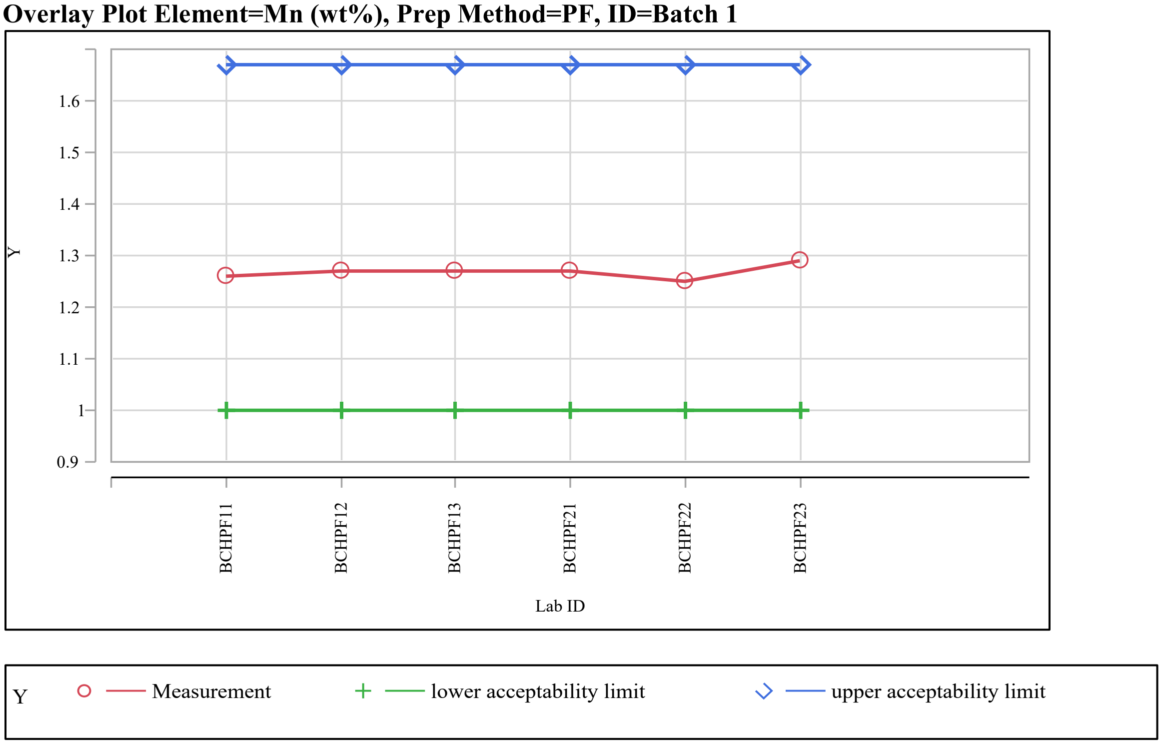
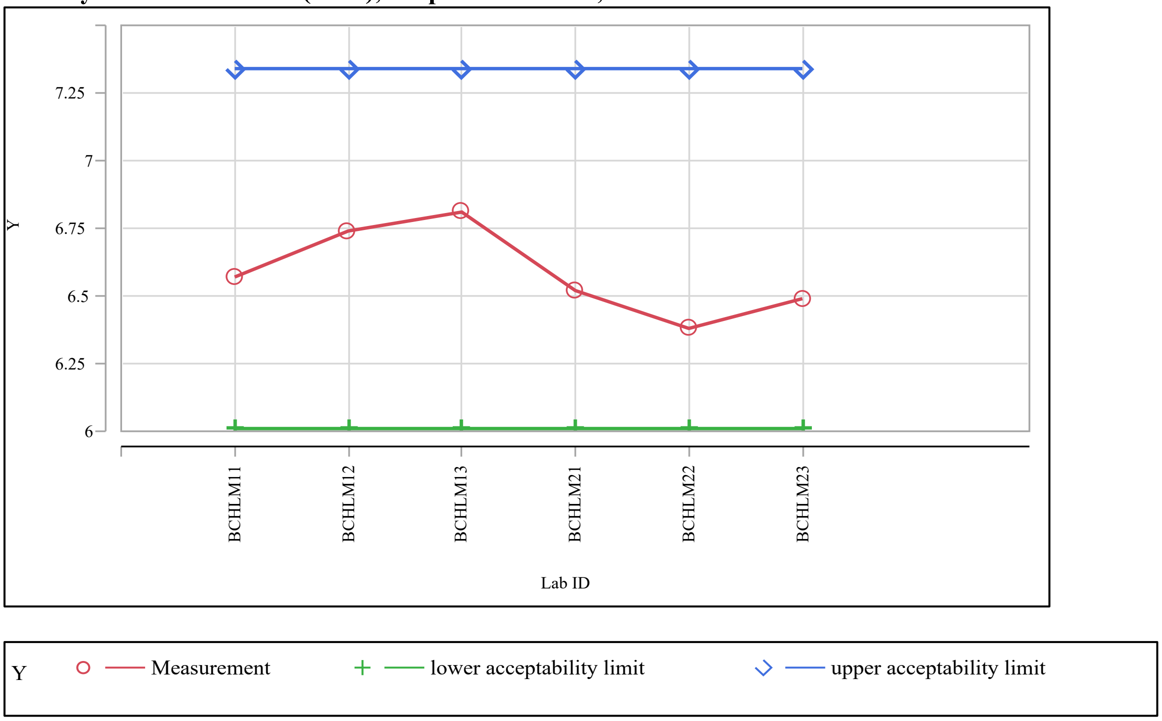


Exhibit A-3. Acceptability Evaluation for Measurements of the Standard Glass (continued)

Overlay Plot Element=Na (wt%), Prep Method=LM, ID=Batch 1



Overlay Plot Element=Ni (wt%), Prep Method=LM, ID=Batch 1

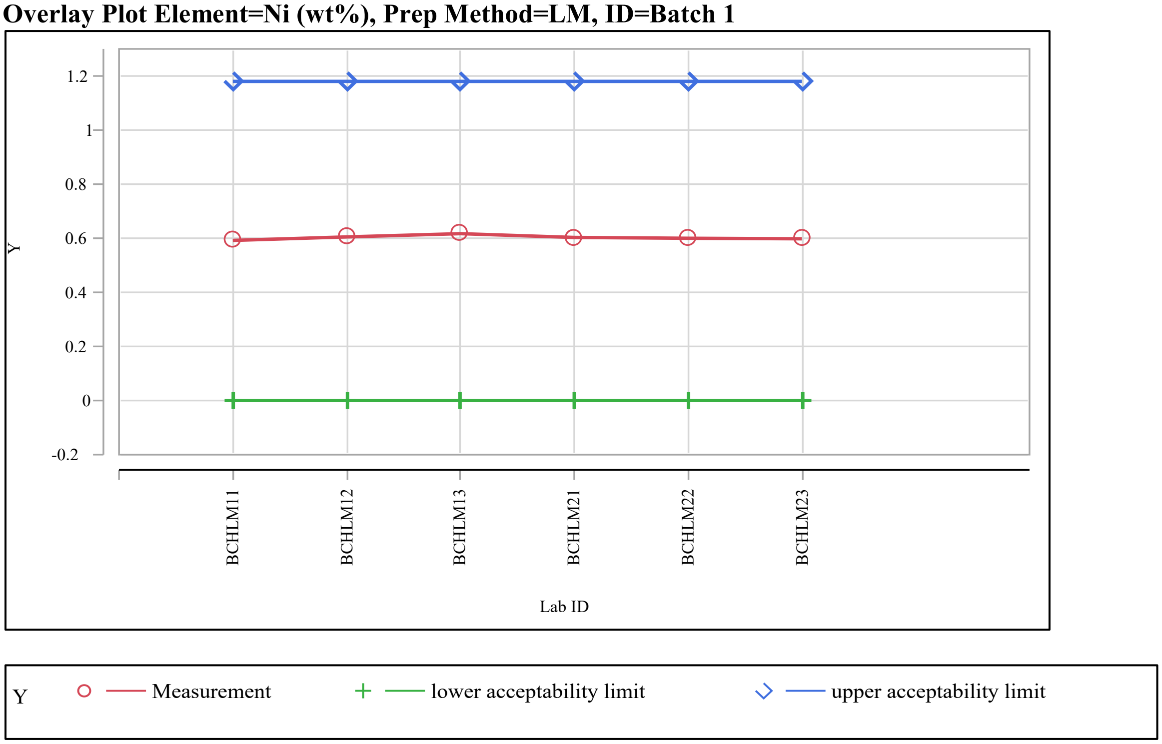
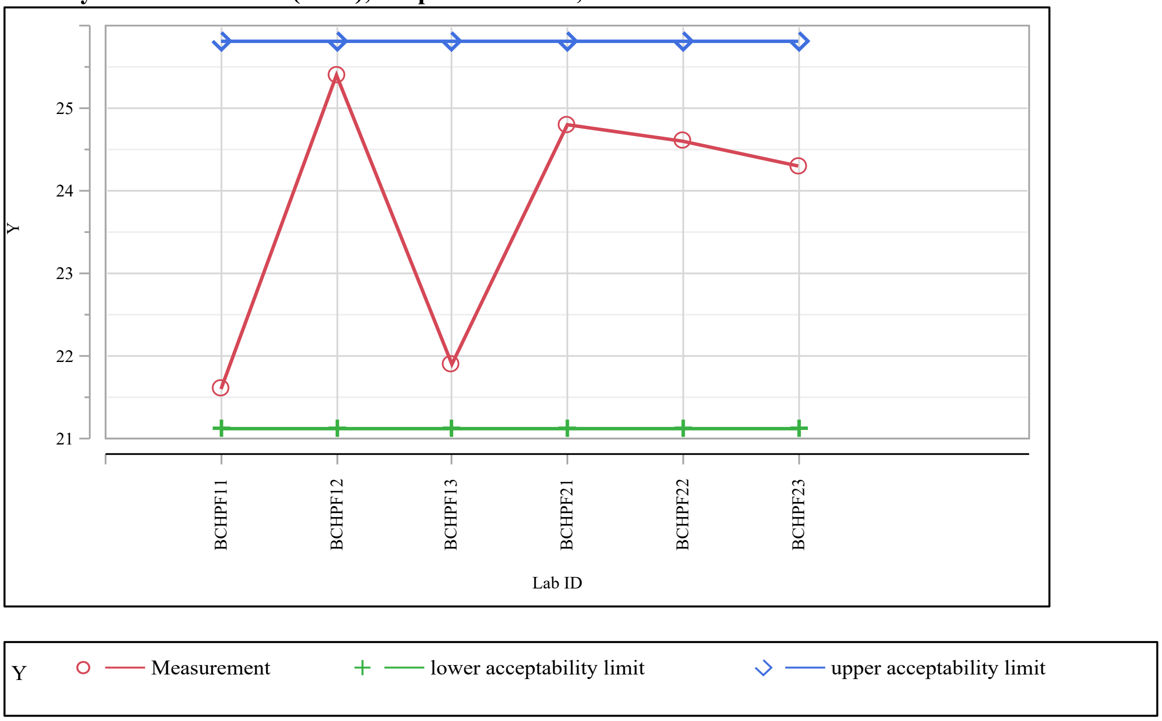


Exhibit A-3. Acceptability Evaluation for Measurements of the Standard Glass (continued)

Overlay Plot Element=Si (wt%), Prep Method=PF, ID=Batch 1



Appendix B Tables and Exhibits Supporting the Viscosity Measurements

Table B-1. Measured Viscosity Data for Glass CJS-2018-2

Time	Sample Temperature (°C)	Viscosity (Poise)	Time	Sample Temperature (°C)	Viscosity (Poise)
10:10	1154	48	13:30	906	914
10:15	1156	48	13:35	902	922
10:20	1157	49	13:40	901	902
10:45	1203	38	13:51	857	1676
10:50	1203	38	13:56	855	1688
10:55	1203	38	14:01	855	1684
11:05	1252	27	14:07	803	4304
11:10	1254	27	14:12	802	4112
11:15	1254	28	14:17	801	4019
11:30	1155	59	14:24	752	12285
11:35	1152	60	14:29	750	11147
11:40	1151	60	14:34	751	10450
11:55	1106	87	15:15	1157	49
12:00	1106	86	15:20	1159	53
12:05	1106	86	15:25	1155	55
12:15	1053	141			
12:20	1051	140			
12:25	1051	139			
12:40	1007	218			
12:45	1005	222			
12:50	1005	245			
13:12	957	400			
13:17	956	437			
13:22	956	481			

Table B-2. Measured Viscosity Data for Glass HLW-HCr-16-X(IV)

Time	Sample Temperature (°C)	Viscosity (Poise)
9:31	1150	58
9:36	1150	59
9:41	1150	60
9:55	1200	43
10:00	1200	43
10:05	1200	44
10:18	1250	31
10:23	1250	32
10:28	1250	32
10:45	1149	68
10:50	1150	67
10:55	1150	67
11:10	1100	101
11:15	1100	100
11:20	1100	100
11:32	1050	160
11:37	1050	157
11:42	1050	157
11:55	1000	261
12:00	1000	257
12:05	1000	255
12:17	950	453
12:22	950	445
12:27	950	445
12:40	900	838
12:45	900	822
12:50	900	811
13:05	850	1682
13:10	850	1649
13:15	851	1625
13:31	800	3984
13:36	800	3894
13:41	800	3813
14:03	750	10410
14:08	750	10267
14:13	750	10063
15:10	1150	67
15:15	1150	67
15:20	1150	68

Table B-3. Measured Viscosity Data for Glass HLW-HCr-16-Y(VI)

Time	Sample Temperature (°C)	Viscosity (Poise)	Time	Sample Temperature (°C)	Viscosity (Poise)
9:36	1150	63	12:42	900	894
9:41	1149	64	12:47	900	869
9:46	1149	65	12:52	900	861
10:05	1200	45	13:10	850	1774
10:10	1200	45	13:15	850	1759
10:15	1200	46	13:20	851	1727
10:25	1250	32	13:35	801	4213
10:30	1251	33	13:40	800	4188
10:35	1250	33	13:45	801	4041
10:45	1150	71	14:00	750	12002
10:50	1148	71	14:05	750	11532
10:55	1150	70	14:10	750	11226
11:07	1100	106	15:00	1151	66
11:12	1100	105	15:05	1150	68
11:17	1100	105	15:10	1150	68
11:32	1050	167			
11:37	1050	165			
11:42	1050	164			
11:52	1001	274			
11:57	1000	271			
12:02	1000	269			
12:20	950	468			
12:25	951	464			
12:30	951	462			

Table B-4. Measured Viscosity Data for Glass HLW-HCr-16-Y(I)

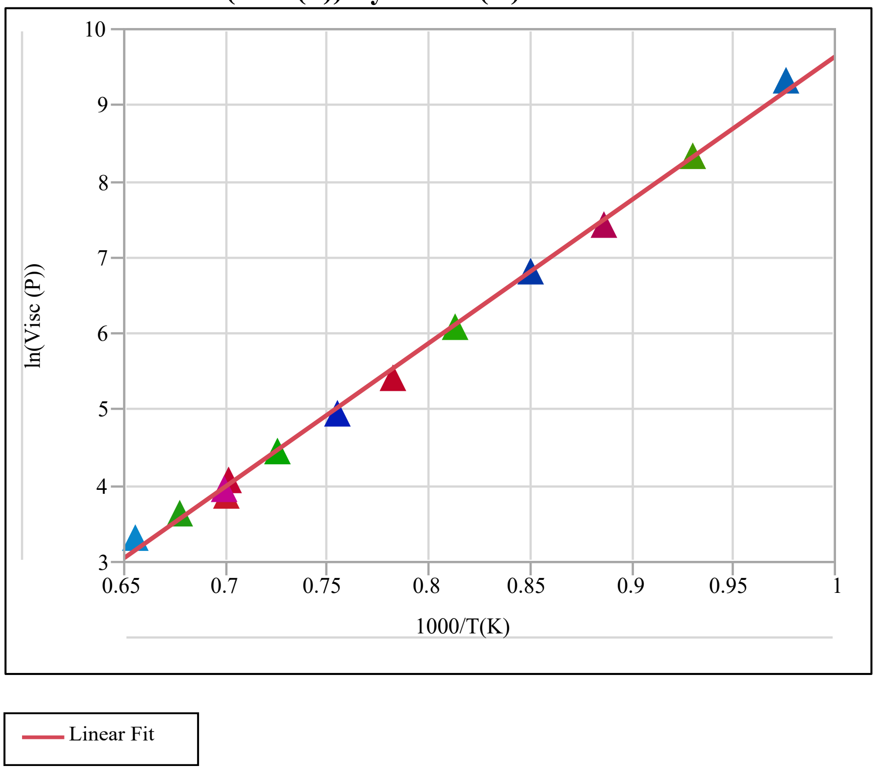
Time	Sample Temperature (°C)	Viscosity (Poise)		Time	Sample Temperature (°C)	Viscosity (Poise)
9:45	1150	70		13:40	900	881
9:50	1150	70		13:45	900	873
9:55	1150	71		13:50	900	866
10:15	1200	51		14:05	850	1760
10:20	1200	51		14:10	850	1737
10:25	1200	52		14:15	850	1709
10:45	1250	38		14:35	800	3838
10:50	1250	38		14:40	800	3754
10:55	1250	38		14:45	800	3726
11:15	1150	80		15:00	750	9853
11:20	1150	79		15:05	750	9643
11:25	1150	79		15:10	751	9479
11:40	1100	119		15:55	1150	73
11:45	1100	117		16:00	1150	75
11:50	1100	117		16:05	1150	76
12:05	1050	182				
12:10	1050	179				
12:15	1050	177				
12:35	1000	290				
12:40	1000	288				
12:45	1000	288				
13:15	950	488				
13:20	950	487				
13:25	950	483				

Table B-5. Measured Viscosity Data for Glass HLW-HCr-16-X(II)

Time	Sample Temperature (°C)	Viscosity (Poise)
9:45	1150	60
9:50	1150	61
9:55	1150	61
10:15	1200	43
10:20	1200	44
10:25	1200	44
10:45	1250	32
10:50	1250	32
10:55	1250	32
11:15	1150	68
11:20	1150	68
11:25	1150	68
11:40	1100	103
11:45	1100	102
11:50	1100	102
12:05	1050	163
12:10	1050	160
12:15	1051	159
12:35	1000	266
12:40	1000	264
12:45	1000	262
13:10	950	463
13:15	950	459
13:20	950	454
13:40	900	843
13:45	901	834
13:50	901	827
14:05	851	1739
14:10	850	1720
14:15	851	1693
14:35	800	4095
14:40	800	3971
14:45	800	3917
15:00	751	11353
15:05	750	11063
15:10	750	10831
15:55	1150	62
16:00	1150	64
16:05	1150	66

Exhibit B-1. Linear Fit of the Average Measured Viscosity Values for Glass CJS-2018-2

Bivariate Fit of ln(Visc (P)) By 1000/T(K) Glass ID=CJS-2018-2



Linear Fit

ln(Visc (P)) = -9.179243 + 18.818348*1000/T(K)

Summary of Fit

RSquare	0.997987
RSquare Adj	0.997804
Root Mean Square Error	0.090899
Mean of Response	5.514795
Observations (or Sum Wgts)	13

Analysis of Variance

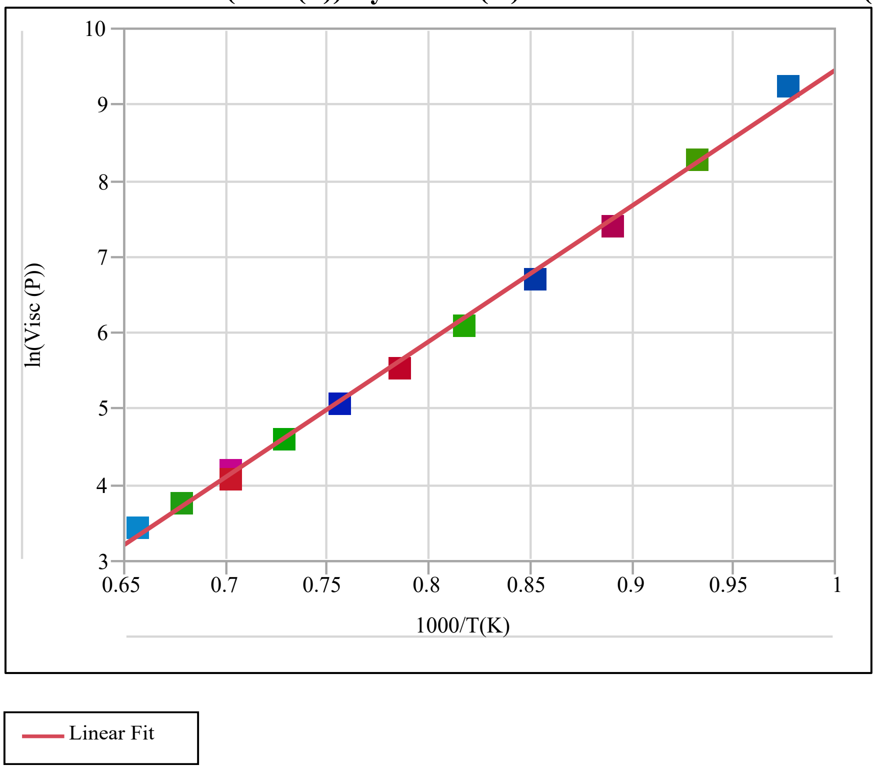
Source	**DF**	**Sum of Squares**	**Mean Square**	**F Ratio**
Model	1	45.066514	45.0665	5454.219
Error	11	0.090890	0.0083	**Prob > F**
C. Total	12	45.157404		<.0001*

Parameter Estimates

Term	**Estimate**	**Std Error**	**t Ratio**	**Prob>|t|**
Intercept	-9.179243	0.200555	-45.77	<.0001*
1000/T(K)	18.818348	0.254809	73.85	<.0001*

Exhibit B-2. Linear Fit of the Average Measured Viscosity Values for Glass HLW-HCr-16-X(IV)

Bivariate Fit of ln(Visc (P)) By 1000/T(K) Glass ID=HLW-HCr-16-X(IV)



Linear Fit

ln(Visc (P)) = -8.371096 + 17.823557*1000/T(K)

Summary of Fit

RSquare	0.997596
RSquare Adj	0.997377
Root Mean Square Error	0.094143
Mean of Response	5.58922
Observations (or Sum Wgts)	13

Analysis of Variance

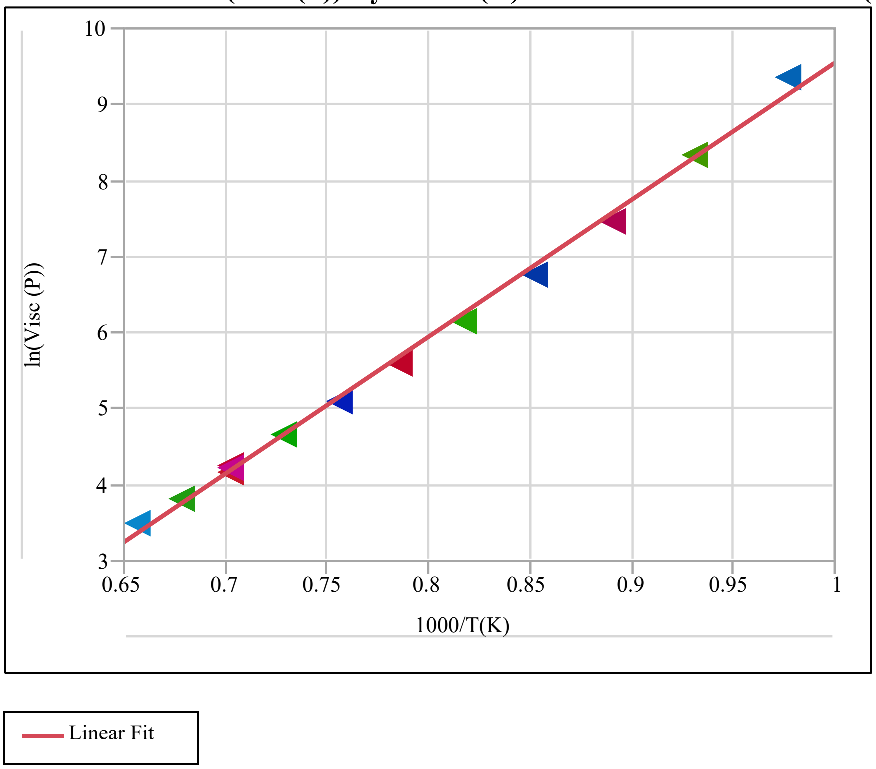
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	40.452773	40.4528	4564.273
Error	11	0.097492	0.0089	**Prob > F**
C. Total	12	40.550265		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-8.371096	0.208281	-40.19	<.0001*
1000/T(K)	17.823557	0.263821	67.56	<.0001*

Exhibit B-3. Linear Fit of the Average Measured Viscosity Values for Glass HLW-HCr-16-Y(VI)

Bivariate Fit of ln(Visc (P)) By 1000/T(K) Glass ID=HLW-HCr-16-Y(VI)



Linear Fit

ln(Visc (P)) = -8.45271 + 17.999519*1000/T(K)

Summary of Fit

RSquare	0.997215
RSquare Adj	0.996962
Root Mean Square Error	0.102253
Mean of Response	5.643982
Observations (or Sum Wgts)	13

Analysis of Variance

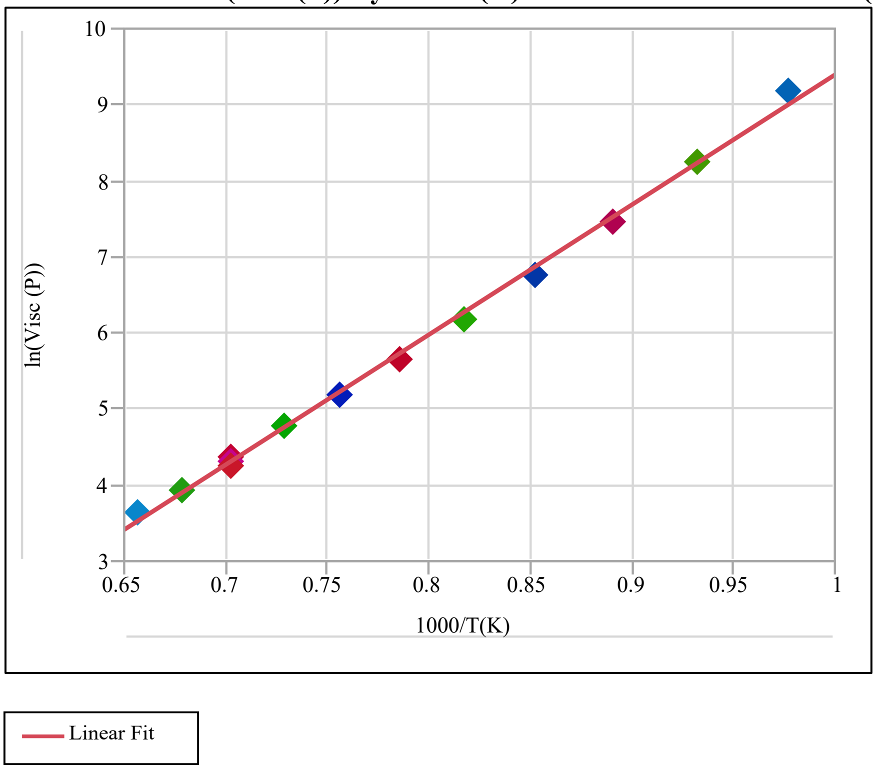
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	41.184381	41.1844	3938.923
Error	11	0.115013	0.0105	**Prob > F**
C. Total	12	41.299395		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-8.45271	0.226393	-37.34	<.0001*
1000/T(K)	17.999519	0.286795	62.76	<.0001*

Exhibit B-4. Linear Fit of the Average Measured Viscosity Values for Glass HLW-HCr-16-Y(I)

Bivariate Fit of ln(Visc (P)) By 1000/T(K) Glass ID=HLW-HCr-16-Y(I)



Linear Fit

ln(Visc (P)) = -7.692354 + 17.08585*1000/T(K)

Summary of Fit

RSquare	0.997854
RSquare Adj	0.997659
Root Mean Square Error	0.085231
Mean of Response	5.689864
Observations (or Sum Wgts)	13

Analysis of Variance

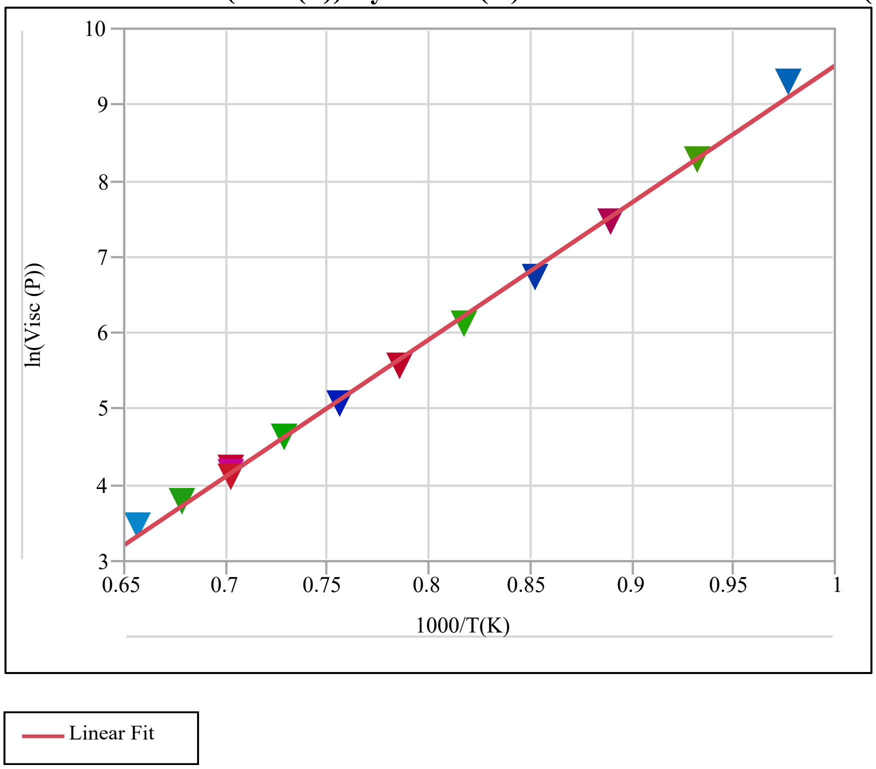
Source	**DF**	**Sum of Squares**	**Mean Square**	**F Ratio**
Model	1	37.161622	37.1616	5115.642
Error	11	0.079907	0.0073	**Prob > F**
C. Total	12	37.241530		<.0001*

Parameter Estimates

Term	**Estimate**	**Std Error**	**t Ratio**	**Prob>|t|**
Intercept	-7.692354	0.188589	-40.79	<.0001*
1000/T(K)	17.08585	0.238884	71.52	<.0001*

Exhibit B-5. Linear Fit of the Average Measured Viscosity Values for Glass HLW-HCr-16-X(II)

Bivariate Fit of ln(Visc (P)) By 1000/T(K) Glass ID=HLW-HCr-16-X(II)



Linear Fit
ln(Visc (P)) = -8.490901 + 18.004289*1000/T(K)

Summary of Fit

RSquare	0.997339
RSquare Adj	0.997097
Root Mean Square Error	0.099982
Mean of Response	5.609004
Observations (or Sum Wgts)	13

Analysis of Variance

Source	**DF**	**Sum of Squares**	**Mean Square**	**F Ratio**
Model	1	41.209340	41.2093	4122.427
Error	11	0.109960	0.0100	**Prob > F**
C. Total	12	41.319300		<.0001*

Parameter Estimates

Term	**Estimate**	**Std Error**	**t Ratio**	**Prob>|t|**
Intercept	-8.490901	0.221348	-38.36	<.0001*
1000/T(K)	18.004289	0.280414	64.21	<.0001*

Appendix C Tables and Exhibits Supporting the PCT Results

Table C-1. PCT Measurements for Round Robin Glasses (ar – as received)

Glass ID	Heat Treatment	Block	Seq	Lab ID	Al ar	B ar	Cr ar	Fe ar	Li ar	Na ar	Si ar
soln std	ref	1	1	std-1-1	3.82	21.1	<1.00	3.79	10.1	82.2	50.3
CJS-2018-2 (as-received) (1st sub-sample)-3	as received	1	2	S-8592	19.7	34.4	<1.00	3.67	13.3	40.3	26.6
CJS-2018-2 (as-received) (2nd sub-sample)-1	as received	1	3	S-8576	19.7	29.5	<1.00	3.88	11.6	37.4	26.2
EA-3	ref	1	4	S-8589	<1.00	33.1	<1.00	<1.00	10.7	96.9	47.9
CJS-2018-2 (CCC) (4th sub-sample)-2	ccc	1	5	S-8590	18.9	35.3	<1.00	2.65	13.4	39.7	26.2
CJS-2018-2 (as-received) (3rd sub-sample)-3	as received	1	6	S-8591	19.7	35.1	<1.00	3.58	13.3	39.9	26.3
soln std	ref	1	7	std-1-2	3.78	19.6	<1.00	3.66	9.99	81.4	46.5
CJS-2018-2 (as-received) (4th sub-sample)-3	as received	1	8	S-8593	19.9	32.0	<1.00	3.87	12.4	38.7	26.1
CJS-2018-2 (CCC) (1st sub-sample)-3	ccc	1	9	S-8597	14.5	59.4	<1.00	1.61	23.9	48.4	27.0
ARM-1-3	ref	1	10	S-8595	3.72	8.06	<1.00	<1.00	7.26	20.6	28.0
CJS-2018-2 (CCC) (2nd sub-sample)-1	ccc	1	11	S-8594	17.7	34.3	<1.00	2.23	13.0	36.1	24.3
CJS-2018-2 (CCC) (3rd sub-sample)-1	ccc	1	12	S-8582	18.2	33.9	<1.00	2.42	12.6	36.5	24.1
blank-2	ref	1	13	S-8584	<1.00	<1.00	<1.00	<1.00	<1.00	1.79	<1.00
soln std	ref	1	14	std-1-3	3.83	19.3	<1.00	3.66	10.1	82.3	45.8
soln std	ref	2	1	std-2-1	4.22	20.6	<1.00	4.17	10.3	80.5	49.6
EA-2	ref	2	2	S-8573	<1.00	33.9	<1.00	<1.00	10.8	92.6	50.3
blank-1	ref	2	3	S-8567	<1.00	<1.00	<1.00	<1.00	<1.00	2.18	<1.00
CJS-2018-2 (CCC) (1st sub-sample)-2	ccc	2	4	S-8596	14.9	59.7	<1.00	2.17	23.4	47.4	31.3
CJS-2018-2 (CCC) (4th sub-sample)-3	ccc	2	5	S-8587	18.8	35.2	<1.00	3.06	13.2	38.1	29.2
ARM-1-2	ref	2	6	S-8580	4.21	9.52	<1.00	<1.00	7.45	20.2	32.1
soln std	ref	2	7	std-2-2	4.23	20.5	<1.00	4.16	10.3	80.4	49.9
CJS-2018-2 (as-received) (3rd sub-sample)-1	as received	2	8	S-8581	19.4	32.3	<1.00	4.04	12.3	36.5	29.1
CJS-2018-2 (CCC) (2nd sub-sample)-3	ccc	2	9	S-8583	17.6	35.6	<1.00	2.74	13.6	36.3	28.5
CJS-2018-2 (as-received) (4th sub-sample)-2	as received	2	10	S-8588	20.2	33.6	<1.00	4.27	12.6	38.3	30.2
CJS-2018-2 (CCC) (3rd sub-sample)-3	ccc	2	11	S-8574	18.8	36.1	<1.00	2.95	13.5	37.8	29.7
CJS-2018-2 (as-received) (2nd sub-sample)-3	as received	2	12	S-8575	19.9	31.1	<1.00	4.34	11.7	36.3	29.9
CJS-2018-2 (as-received) (1st sub-sample)-2	as received	2	13	S-8579	20.0	36.7	<1.00	4.13	13.6	39.8	30.0
soln std	ref	2	14	std-2-3	4.27	20.8	<1.00	4.16	10.3	79.6	50.1
soln std	ref	3	1	std-3-1	3.91	20.5	<1.00	3.88	10.2	82.1	50.5
CJS-2018-2 (CCC) (4th sub-sample)-1	ccc	3	2	S-8586	18.5	34.3	<1.00	2.73	12.8	37.5	29.1
EA-1	ref	3	3	S-8569	<1.00	32.8	<1.00	<1.00	10.7	92.5	51.5
CJS-2018-2 (CCC) (2nd sub-sample)-2	ccc	3	4	S-8578	17.7	36.4	<1.00	2.38	13.5	37.3	28.8
CJS-2018-2 (as-received) (3rd sub-sample)-2	as received	3	5	S-8571	19.2	32.9	<1.00	3.54	12.4	37.3	29.1
CJS-2018-2 (CCC) (3rd sub-sample)-2	ccc	3	6	S-8566	18.0	33.6	<1.00	2.48	12.5	36.1	28.7
soln std	ref	3	7	std-3-2	3.88	19.9	<1.00	3.76	10.0	81.1	49.7
CJS-2018-2 (as-received) (4th sub-sample)-1	as received	3	8	S-8572	19.4	31.5	<1.00	3.75	12.1	37.6	29.0
CJS-2018-2 (CCC) (1st sub-sample)-1	ccc	3	9	S-8585	14.3	58.4	<1.00	1.69	23.3	47.1	30.6
CJS-2018-2 (as-received) (2nd sub-sample)-2	as received	3	10	S-8568	19.5	28.8	<1.00	3.88	11.3	36.5	29.2
CJS-2018-2 (as-received) (1st sub-sample)-1	as received	3	11	S-8577	20.1	36.5	<1.00	3.73	13.5	41.0	29.9
ARM-1-1	ref	3	12	S-8570	3.99	8.87	<1.00	<1.00	7.2	20.0	31.8
soln std	ref	3	13	std-3-3	3.92	20.1	<1.00	3.78	10.1	81.5	49.7

Table C-2. PCT Measurements for Round Robin Glasses, Corrected for Dilutions

Glass ID	Heat Treatment	Block	Seq	Lab ID	Al (mg/L)	B (mg/L)	Cr (mg/L)	Fe (mg/L)	Li (mg/L)	Na (mg/L)	Si (mg/L)
soln std	ref	1	1	std-1-1	3.820	21.100	1.000	3.790	10.100	82.200	50.300
CJS-2018-2 (as-received) (1st sub-sample)-3	as received	1	2	S-8592	32.834	57.334	1.667	6.117	22.167	67.168	44.334
CJS-2018-2 (as-received) (2nd sub-sample)-1	as received	1	3	S-8576	32.834	49.168	1.667	6.467	19.334	62.335	43.668
EA-3	ref	1	4	S-8589	16.667	551.668	16.667	16.667	178.334	1615.003	798.335
CJS-2018-2 (CCC) (4th sub-sample)-2	ccc	1	5	S-8590	31.501	58.835	1.667	4.417	22.334	66.168	43.668
CJS-2018-2 (as-received) (3rd sub-sample)-3	as received	1	6	S-8591	32.834	58.501	1.667	5.967	22.167	66.501	43.834
soln std	ref	1	7	std-1-2	3.780	19.600	1.000	3.660	9.990	81.400	46.500
CJS-2018-2 (as-received) (4th sub-sample)-3	as received	1	8	S-8593	33.167	53.334	1.667	6.450	20.667	64.501	43.501
CJS-2018-2 (CCC) (1st sub-sample)-3	ccc	1	9	S-8597	24.167	99.002	1.667	2.683	39.834	80.668	45.001
ARM-1-3	ref	1	10	S-8595	6.200	13.434	1.667	1.667	12.100	34.334	46.668
CJS-2018-2 (CCC) (2nd sub-sample)-1	ccc	1	11	S-8594	29.501	57.168	1.667	3.717	21.667	60.168	40.501
CJS-2018-2 (CCC) (3rd sub-sample)-1	ccc	1	12	S-8582	30.334	56.501	1.667	4.033	21.000	60.835	40.167
blank-2	ref	1	13	S-8584	1.667	1.667	1.667	1.667	1.667	2.983	1.667
soln std	ref	1	14	std-1-3	3.830	19.300	1.000	3.660	10.100	82.300	45.800
soln std	ref	2	1	std-2-1	4.220	20.600	1.000	4.170	10.300	80.500	49.600
EA-2	ref	2	2	S-8573	16.667	565.001	16.667	16.667	180.000	1543.336	838.335
blank-1	ref	2	3	S-8567	1.667	1.667	1.667	1.667	1.667	3.633	1.667
CJS-2018-2 (CCC) (1st sub-sample)-2	ccc	2	4	S-8596	24.834	99.502	1.667	3.617	39.001	79.002	52.168
CJS-2018-2 (CCC) (4th sub-sample)-3	ccc	2	5	S-8587	31.334	58.668	1.667	5.100	22.000	63.501	48.668
ARM-1-2	ref	2	6	S-8580	7.017	15.867	1.667	1.667	12.417	33.667	53.501
soln std	ref	2	7	std-2-2	4.230	20.500	1.000	4.160	10.300	80.400	49.900
CJS-2018-2 (as-received) (3rd sub-sample)-1	as received	2	8	S-8581	32.334	53.834	1.667	6.733	20.500	60.835	48.501
CJS-2018-2 (CCC) (2nd sub-sample)-3	ccc	2	9	S-8583	29.334	59.335	1.667	4.567	22.667	60.501	47.501
CJS-2018-2 (as-received) (4th sub-sample)-2	as received	2	10	S-8588	33.667	56.001	1.667	7.117	21.000	63.835	50.334
CJS-2018-2 (CCC) (3rd sub-sample)-3	ccc	2	11	S-8574	31.334	60.168	1.667	4.917	22.500	63.001	49.501
CJS-2018-2 (as-received) (2nd sub-sample)-3	as received	2	12	S-8575	33.167	51.834	1.667	7.233	19.500	60.501	49.834
CJS-2018-2 (as-received) (1st sub-sample)-2	as received	2	13	S-8579	33.334	61.168	1.667	6.883	22.667	66.335	50.001
soln std	ref	2	14	std-2-3	4.270	20.800	1.000	4.160	10.300	79.600	50.100
soln std	ref	3	1	std-3-1	3.910	20.500	1.000	3.880	10.200	82.100	50.500
CJS-2018-2 (CCC) (4th sub-sample)-1	ccc	3	2	S-8586	30.834	57.168	1.667	4.550	21.334	62.501	48.501
EA-1	ref	3	3	S-8569	16.667	546.668	16.667	16.667	178.334	1541.670	858.335
CJS-2018-2 (CCC) (2nd sub-sample)-2	ccc	3	4	S-8578	29.501	60.668	1.667	3.967	22.500	62.168	48.001
CJS-2018-2 (as-received) (3rd sub-sample)-2	as received	3	5	S-8571	32.001	54.834	1.667	5.900	20.667	62.168	48.501
CJS-2018-2 (CCC) (3rd sub-sample)-2	ccc	3	6	S-8566	30.001	56.001	1.667	4.133	20.834	60.168	47.834
soln std	ref	3	7	std-3-2	3.880	19.900	1.000	3.760	10.000	81.100	49.700
CJS-2018-2 (as-received) (4th sub-sample)-1	as received	3	8	S-8572	32.334	52.501	1.667	6.250	20.167	62.668	48.334
CJS-2018-2 (CCC) (1st sub-sample)-1	ccc	3	9	S-8585	23.834	97.335	1.667	2.817	38.834	78.502	51.001
CJS-2018-2 (as-received) (2nd sub-sample)-2	as received	3	10	S-8568	32.501	48.001	1.667	6.467	18.834	60.835	48.668
CJS-2018-2 (as-received) (1st sub-sample)-1	as received	3	11	S-8577	33.501	60.835	1.667	6.217	22.500	68.335	49.834
ARM-1-1	ref	3	12	S-8570	6.650	14.784	1.667	1.667	12.000	33.334	53.001
soln std	ref	3	13	std-3-3	3.920	20.100	1.000	3.780	10.100	81.500	49.700

Table C-3. PCT Leachate pH Values

Identifier	pH		Identifier	pH
ARM-1-1	10.31		CJS-2018-2 (as-received) (4th sub-sample)-1	9.83
ARM-1-2	10.38		CJS-2018-2 (as-received) (4th sub-sample)-2	9.88
ARM-1-3	10.19		CJS-2018-2 (as-received) (4th sub-sample)-3	9.90
blank-1	6.77		CJS-2018-2 (CCC) (1st sub-sample)-1	9.92
blank-2	5.80		CJS-2018-2 (CCC) (1st sub-sample)-2	10.24
EA-1	11.63		CJS-2018-2 (CCC) (1st sub-sample)-3	10.04
EA-2	11.82		CJS-2018-2 (CCC) (2nd sub-sample)-1	9.77
EA-3	11.83		CJS-2018-2 (CCC) (2nd sub-sample)-2	9.93
CJS-2018-2 (as-received) (1st sub-sample)-1	9.97		CJS-2018-2 (CCC) (2nd sub-sample)-3	9.90
CJS-2018-2 (as-received) (1st sub-sample)-2	10.05		CJS-2018-2 (CCC) (3rd sub-sample)-1	9.77
CJS-2018-2 (as-received) (1st sub-sample)-3	10.08		CJS-2018-2 (CCC) (3rd sub-sample)-2	10.07
CJS-2018-2 (as-received) (2nd sub-sample)-1	9.90		CJS-2018-2 (CCC) (3rd sub-sample)-3	9.98
CJS-2018-2 (as-received) (2nd sub-sample)-2	10.13		CJS-2018-2 (CCC) (4th sub-sample)-1	9.84
CJS-2018-2 (as-received) (2nd sub-sample)-3	10.03		CJS-2018-2 (CCC) (4th sub-sample)-2	9.92
CJS-2018-2 (as-received) (3rd sub-sample)-1	10.10		CJS-2018-2 (CCC) (4th sub-sample)-3	10.22
CJS-2018-2 (as-received) (3rd sub-sample)-2	10.04			
CJS-2018-2 (as-received) (3rd sub-sample)-3	10.12			

Exhibit C-1. PCT Measurements in Analytical Sequence by Analytical Block

Analyte=Al
Variability Chart for log (Measured mg/L)

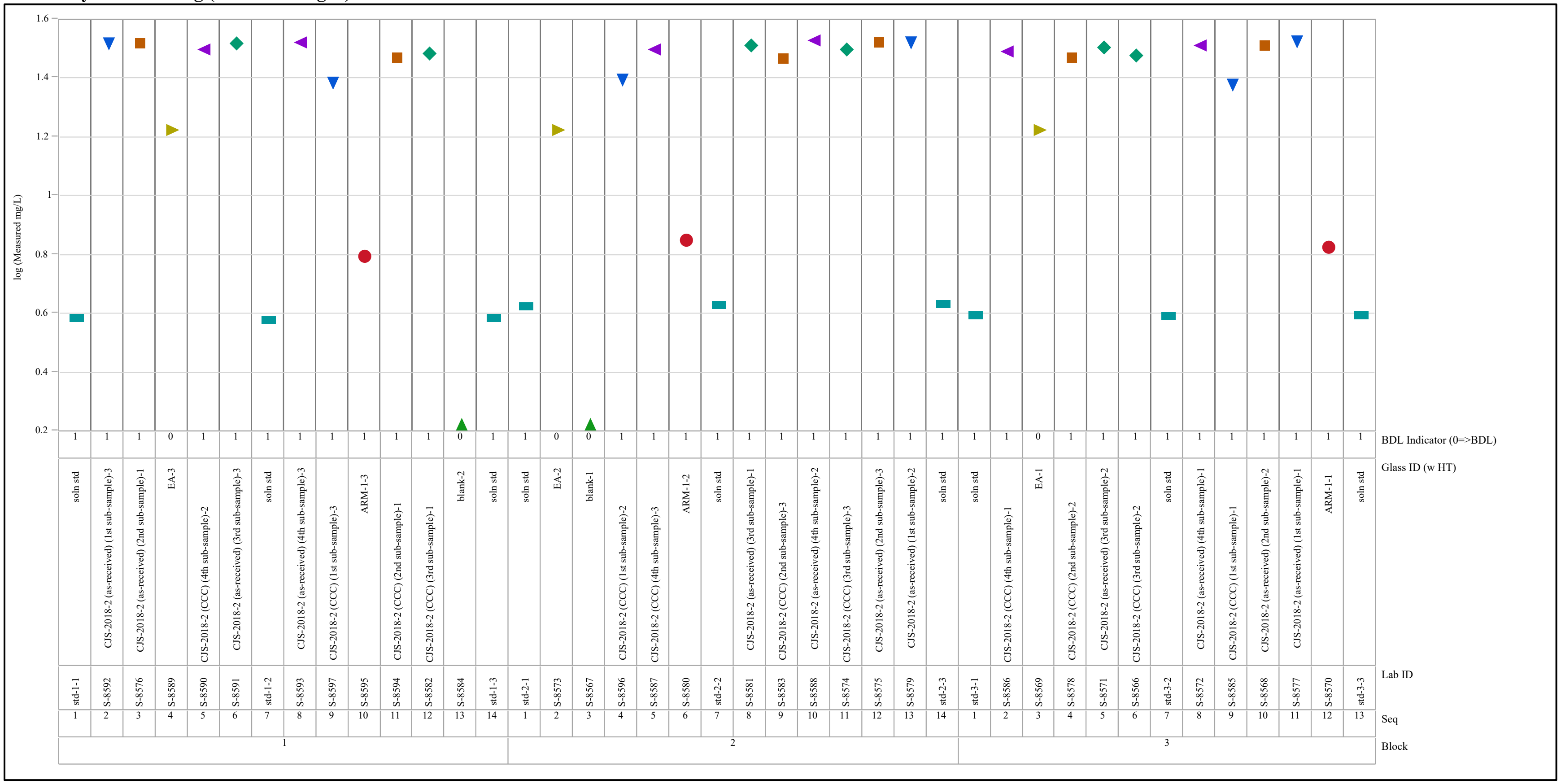


Exhibit C-1. PCT Measurements in Analytical Sequence by Analytical Block (continued)

Analyte=B
Variability Chart for log (Measured mg/L)

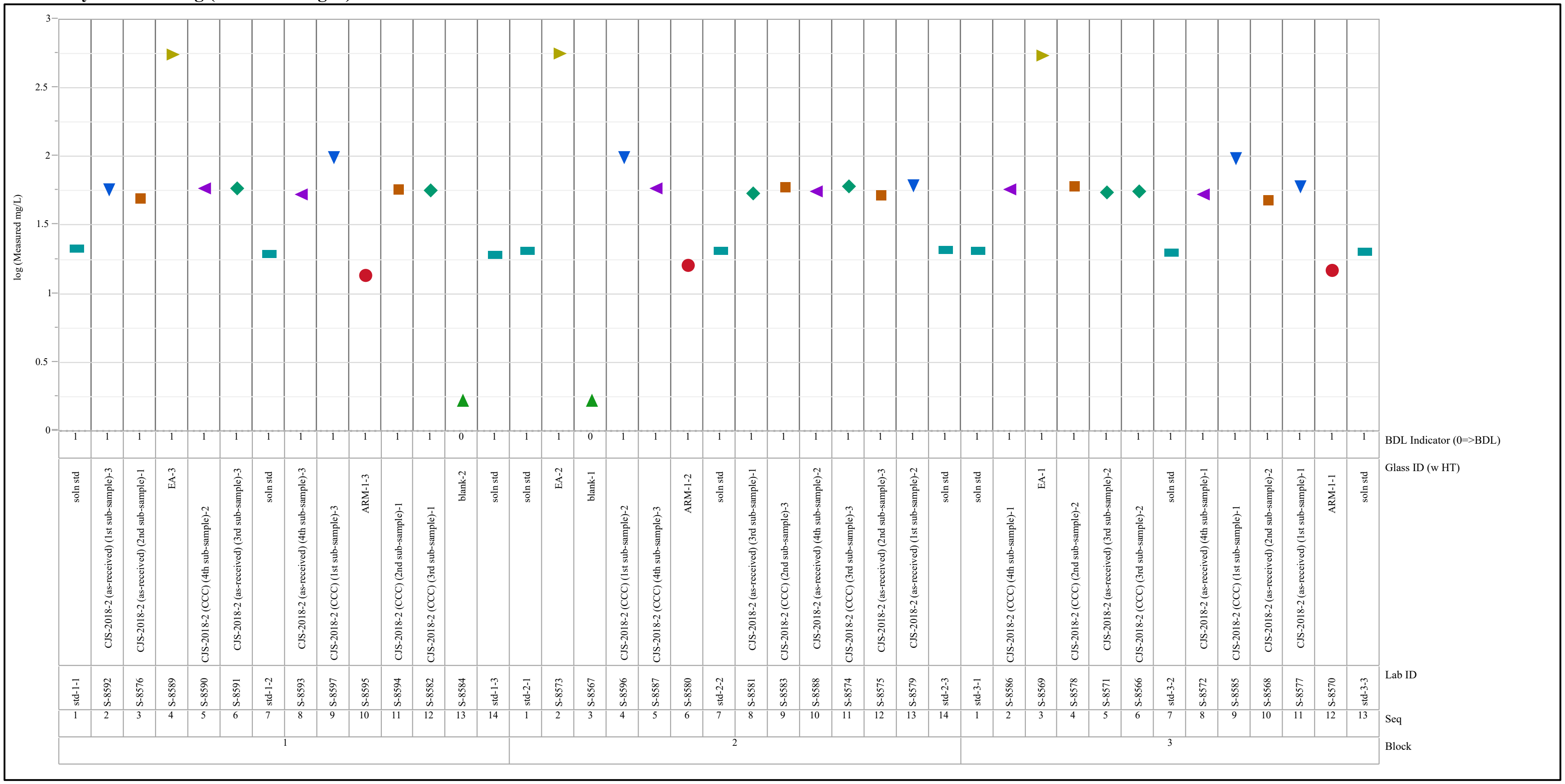


Exhibit C-1. PCT Measurements in Analytical Sequence by Analytical Block (continued)

Analyte=Cr
Variability Chart for log (Measured mg/L)

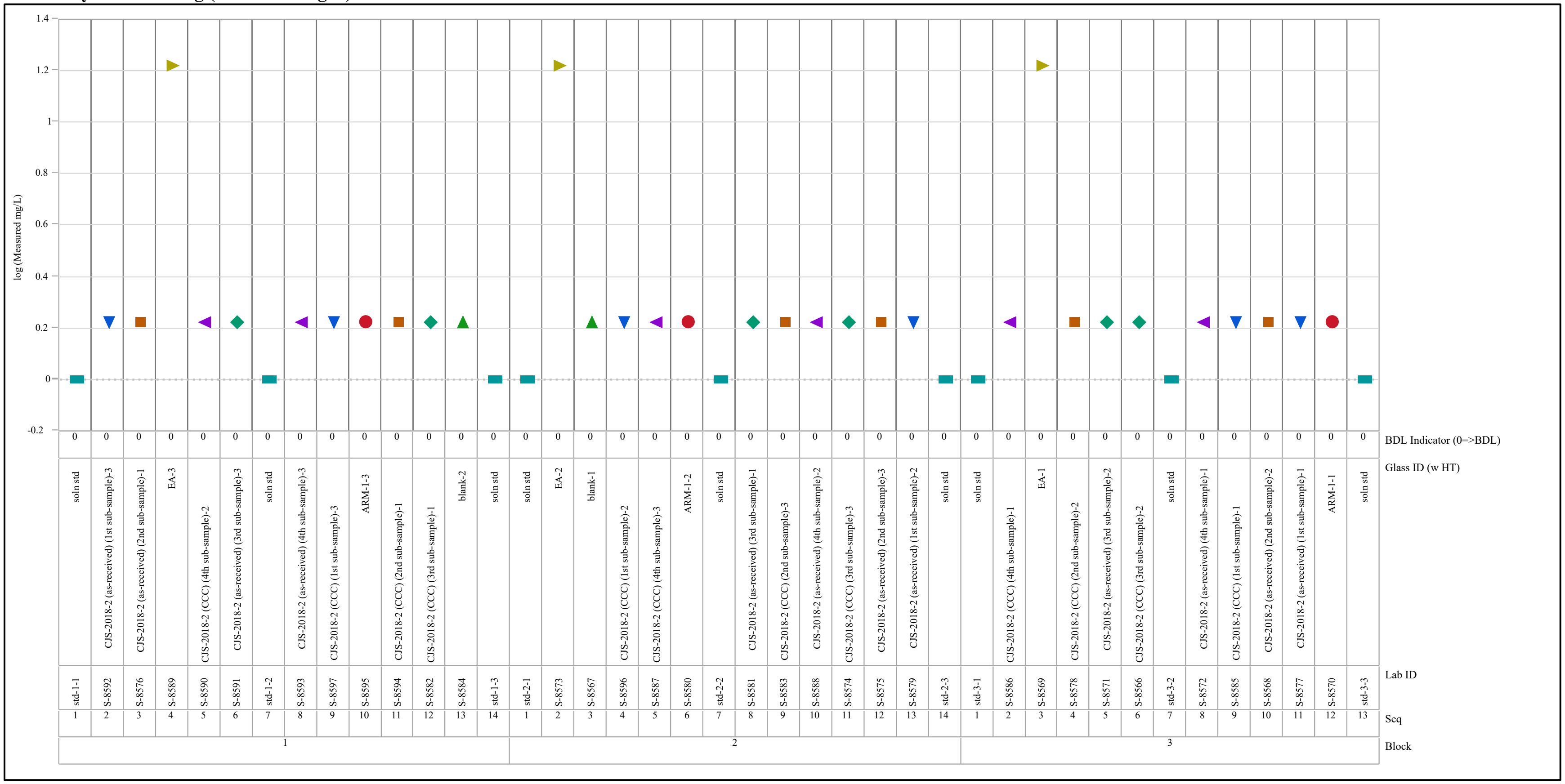


Exhibit C-1. PCT Measurements in Analytical Sequence by Analytical Block (continued)

Analyte=Fe
Variability Chart for log (Measured mg/L)

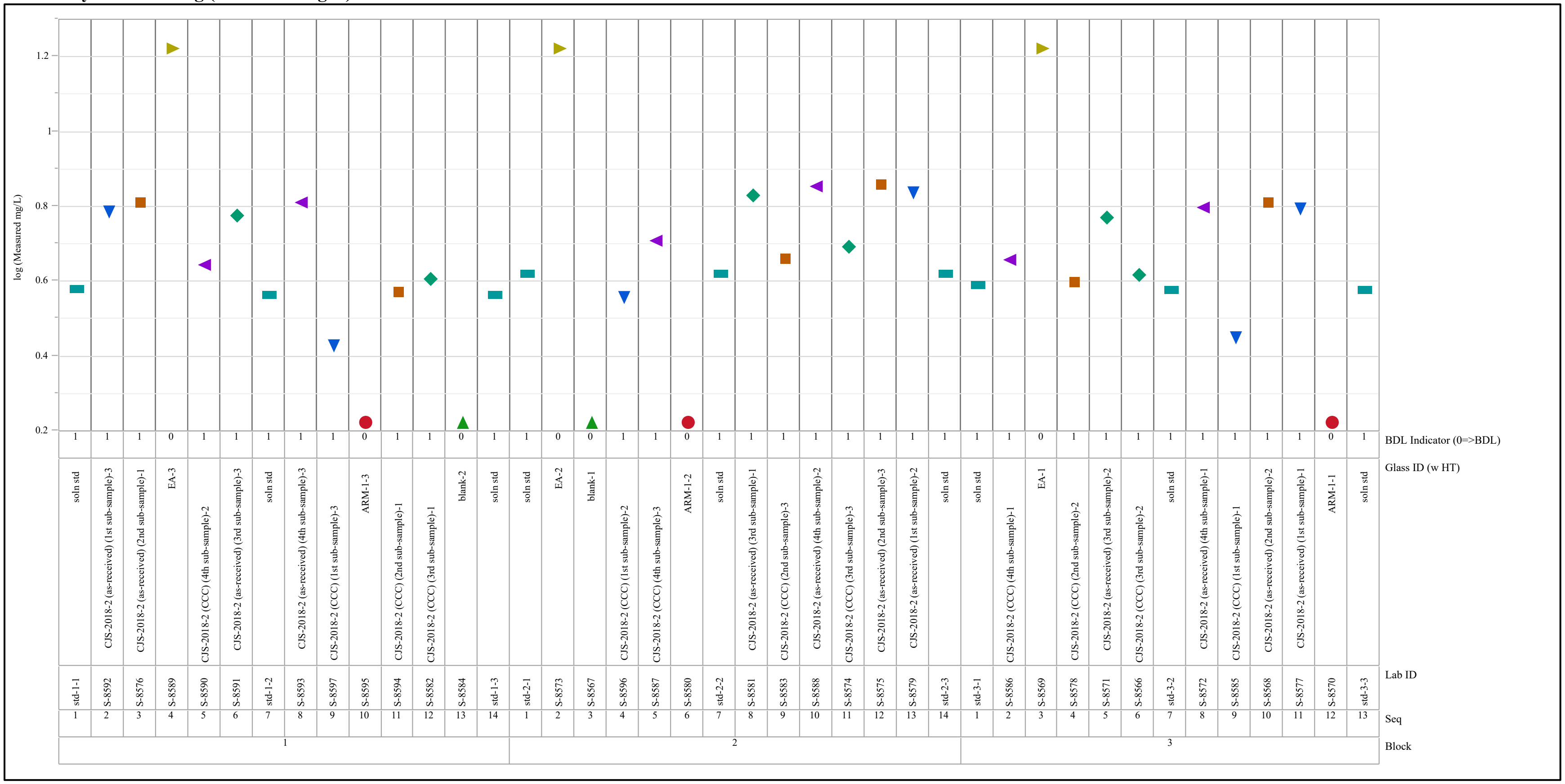


Exhibit C-1. PCT Measurements in Analytical Sequence by Analytical Block (continued)

Analyte=Li
Variability Chart for log (Measured mg/L)

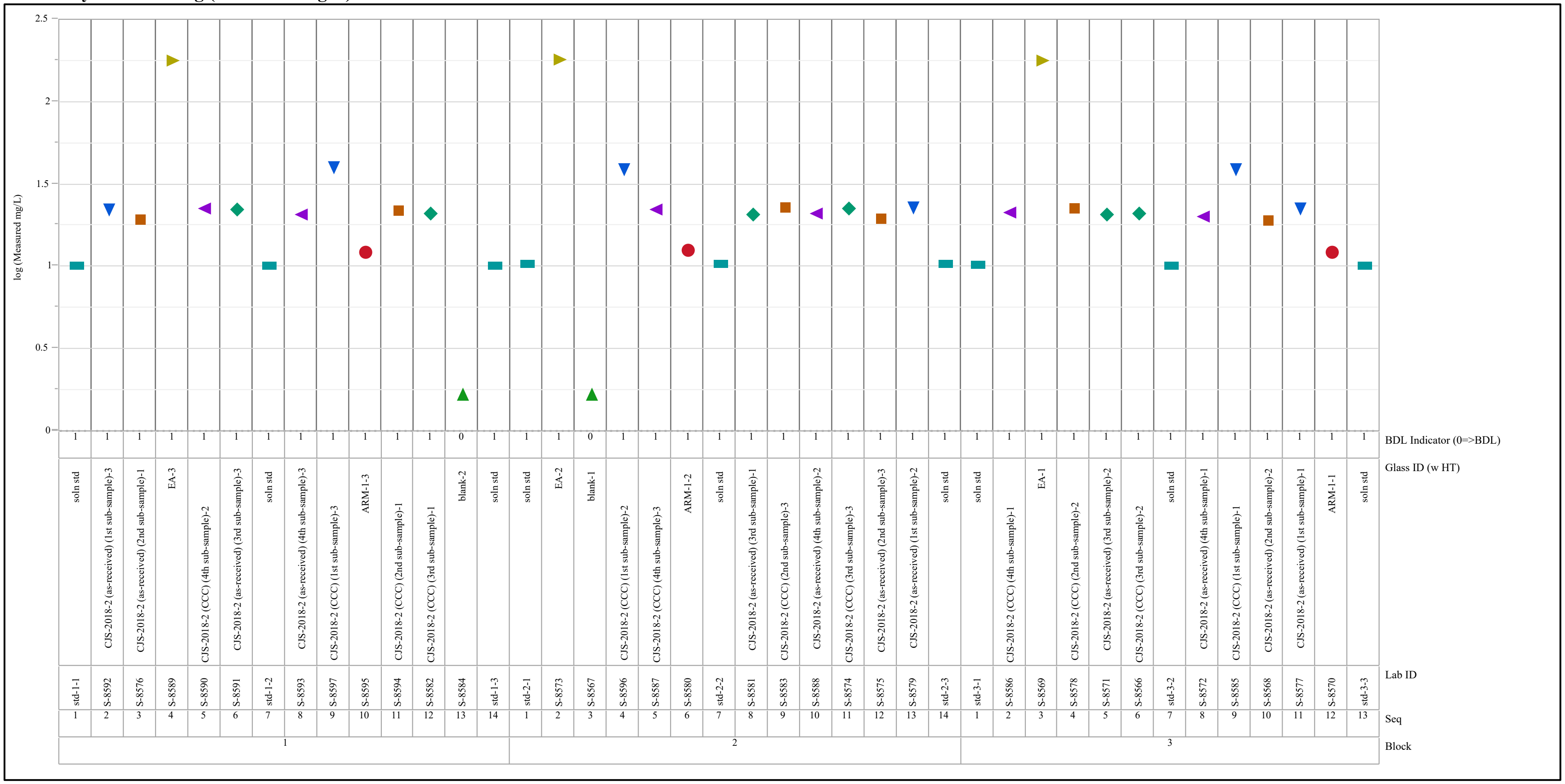


Exhibit C-1. PCT Measurements in Analytical Sequence by Analytical Block (continued)

Analyte=Na
Variability Chart for log (Measured mg/L)

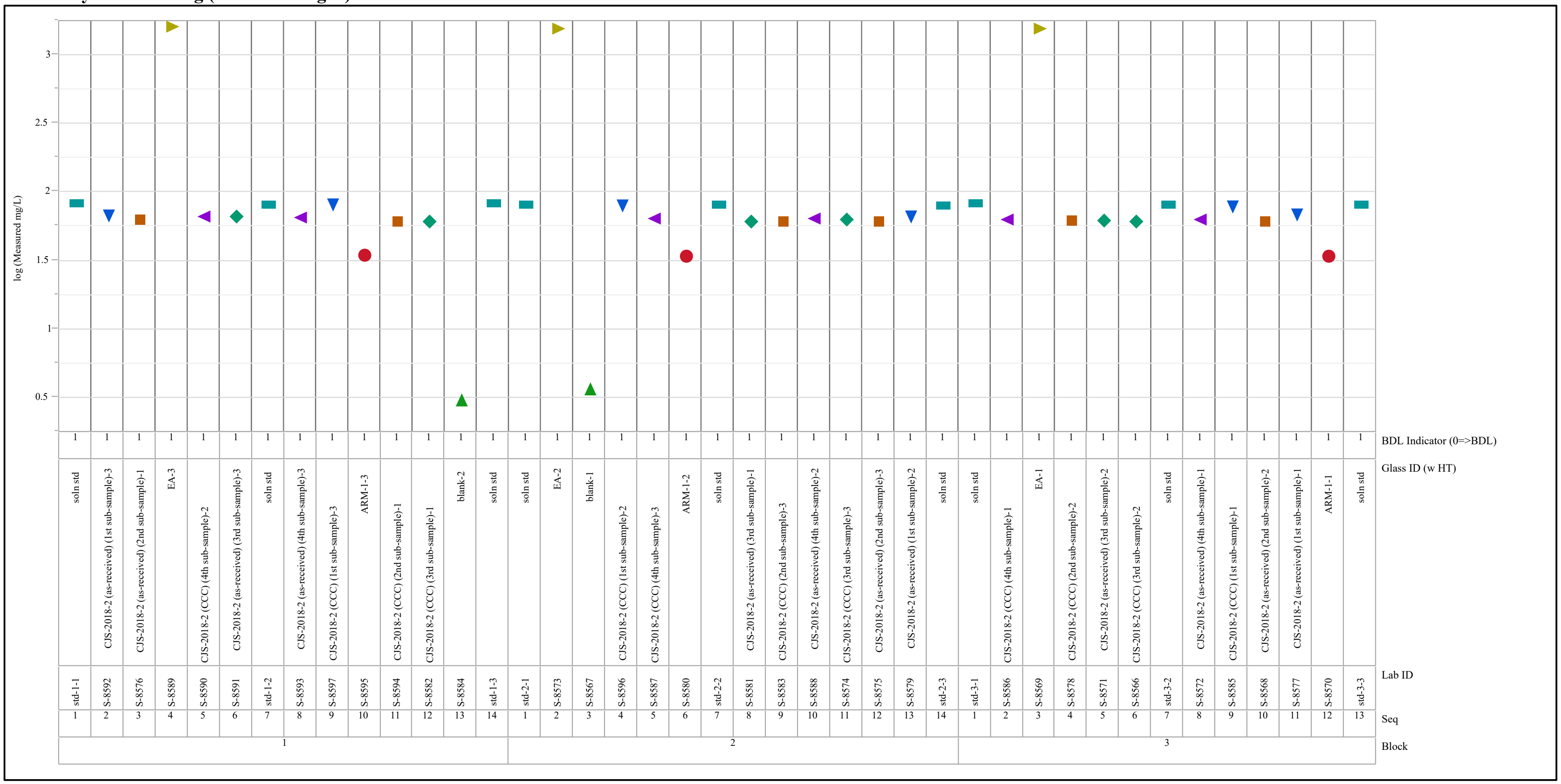


Exhibit C-1. PCT Measurements in Analytical Sequence by Analytical Block (continued)

Analyte=Si
Variability Chart for log (Measured mg/L)

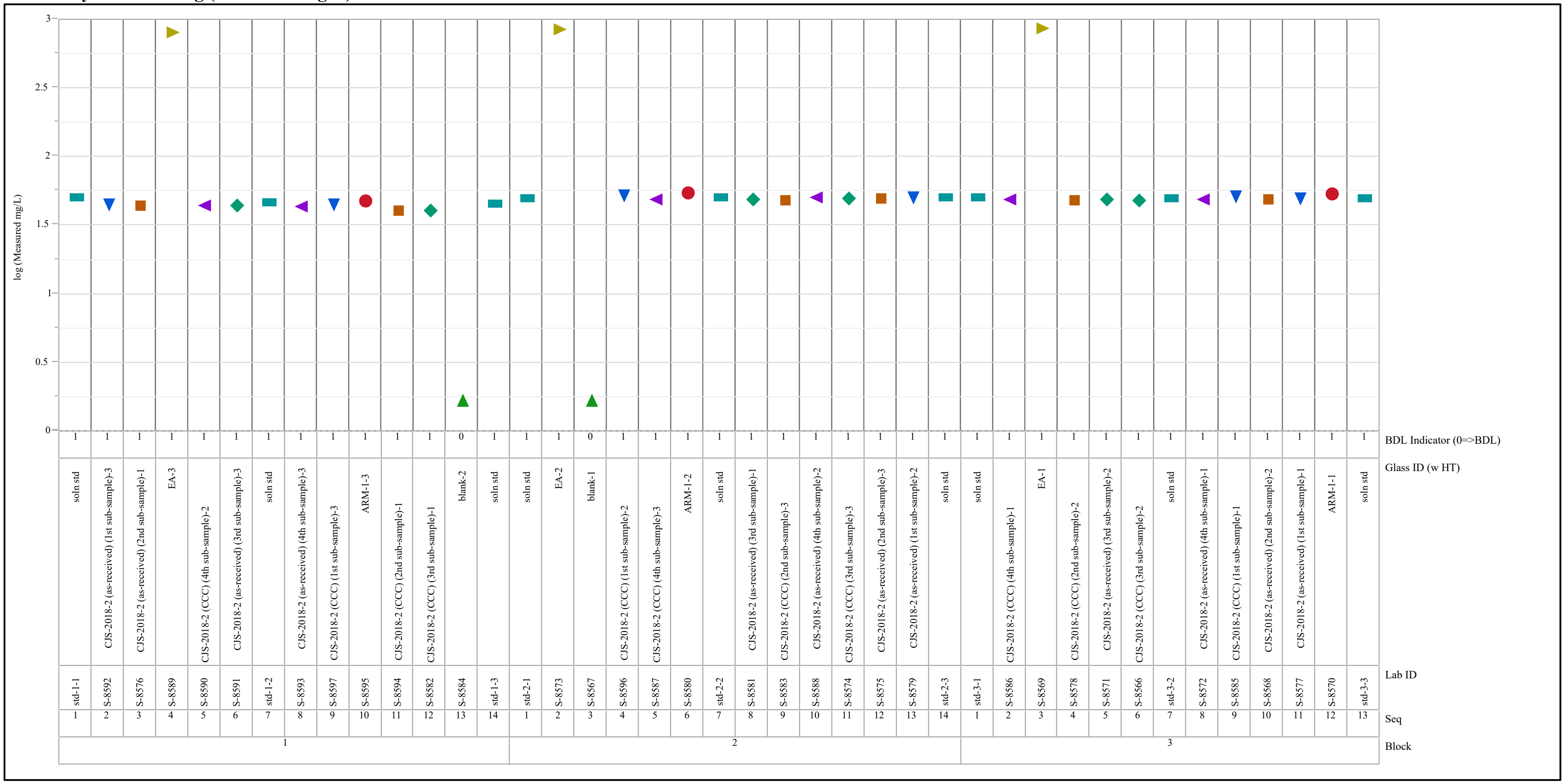


Exhibit C-2. PCT Measurements for Each Glass Subsample Grouped by Heat Treatment

Analyte=Al
Variability Chart for log (Measured mg/L)

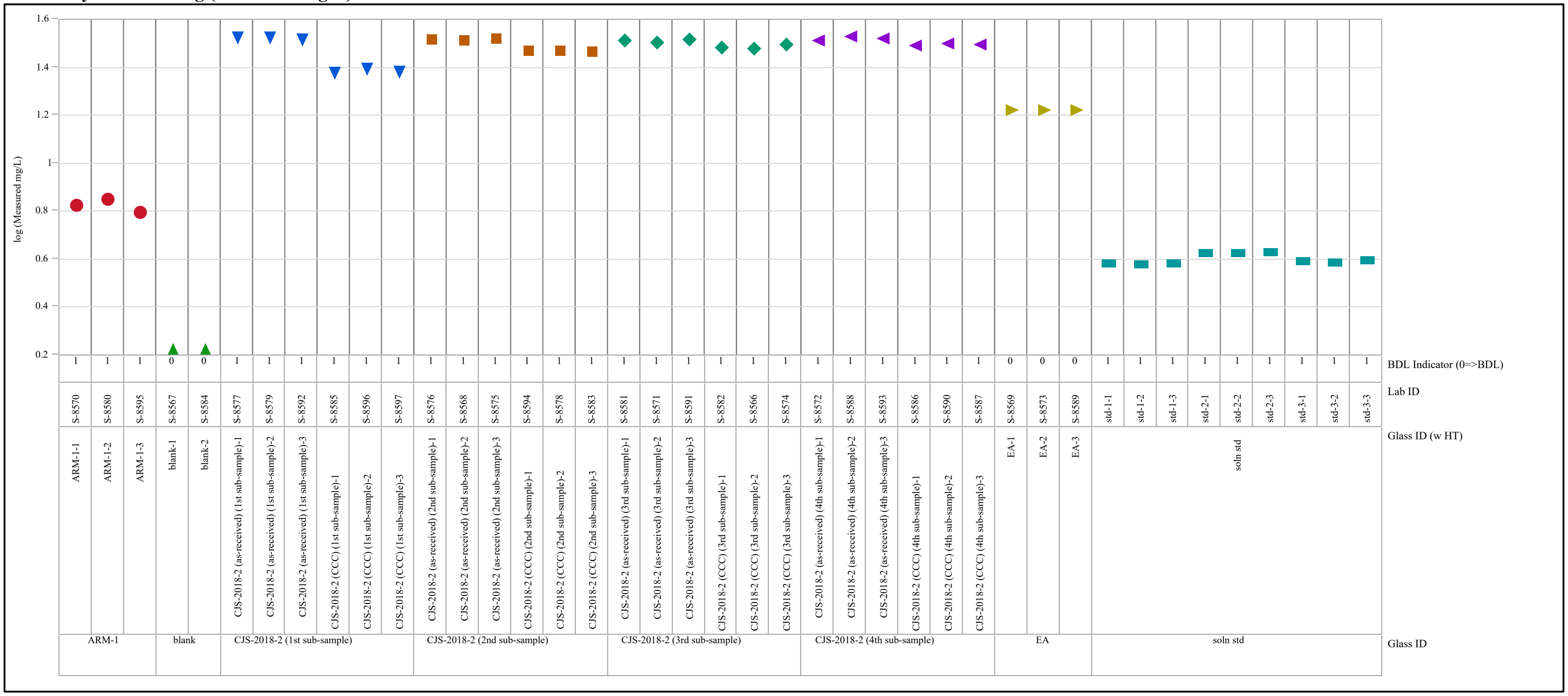


Exhibit C-2. PCT Measurements for Each Glass Subsample Grouped by Heat Treatment (continued)

Analyte=B
Variability Chart for log (Measured mg/L)

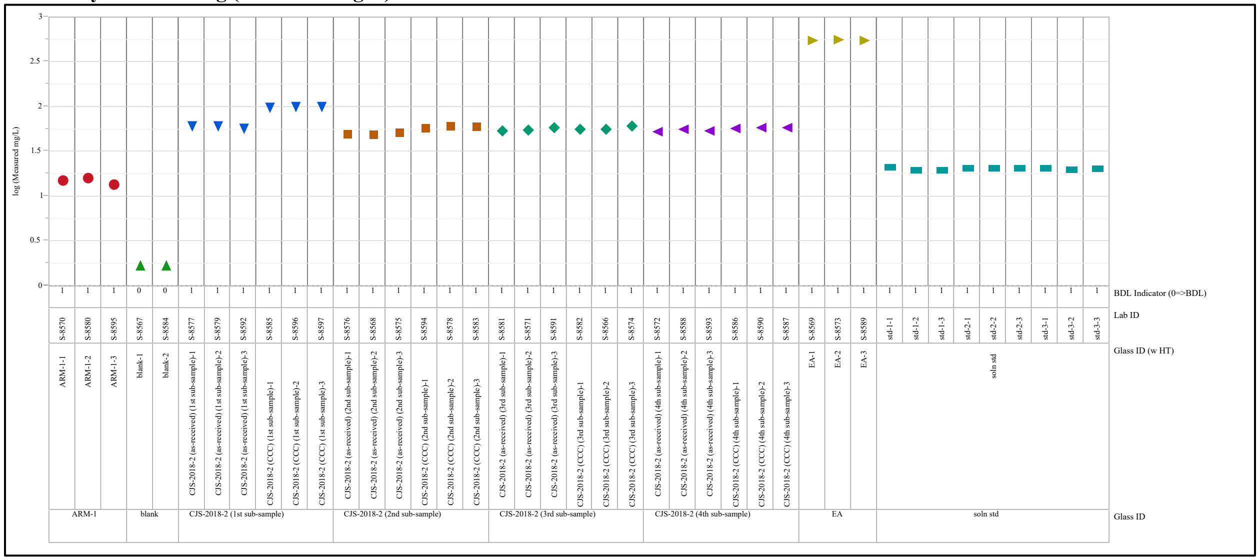


Exhibit C-2. PCT Measurements for Each Glass Subsample Grouped by Heat Treatment (continued)

Analyte=Cr
Variability Chart for log (Measured mg/L)

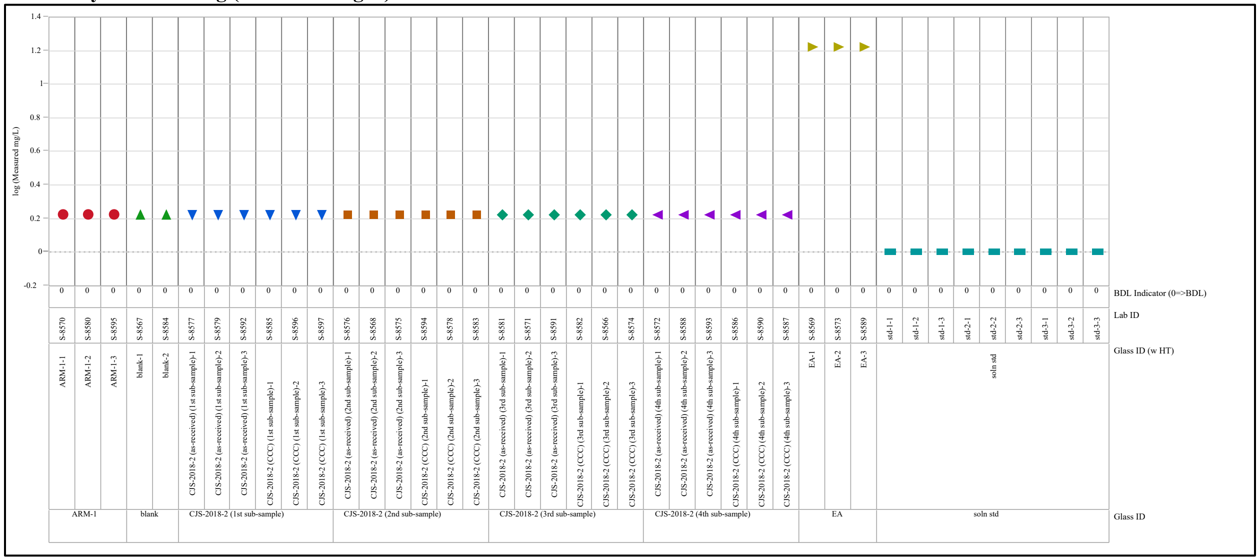


Exhibit C-2. PCT Measurements for Each Glass Subsample Grouped by Heat Treatment (continued)

Analyte=Fe
Variability Chart for log (Measured mg/L)

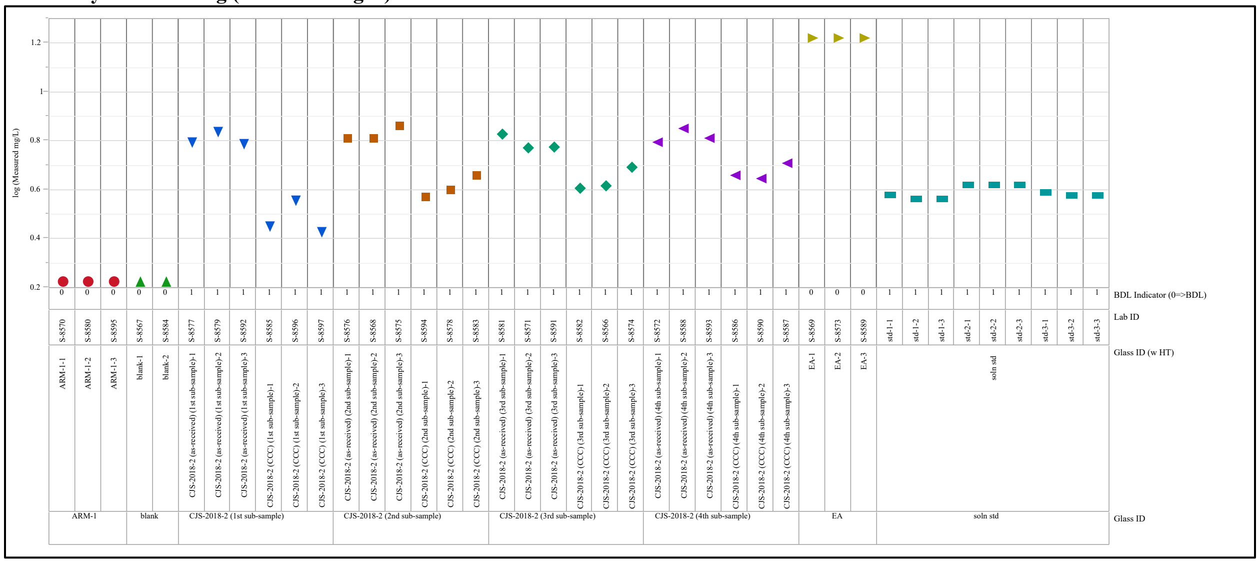


Exhibit C-2. PCT Measurements for Each Glass Subsample Grouped by Heat Treatment (continued)

Analyte=Li
Variability Chart for log (Measured mg/L)

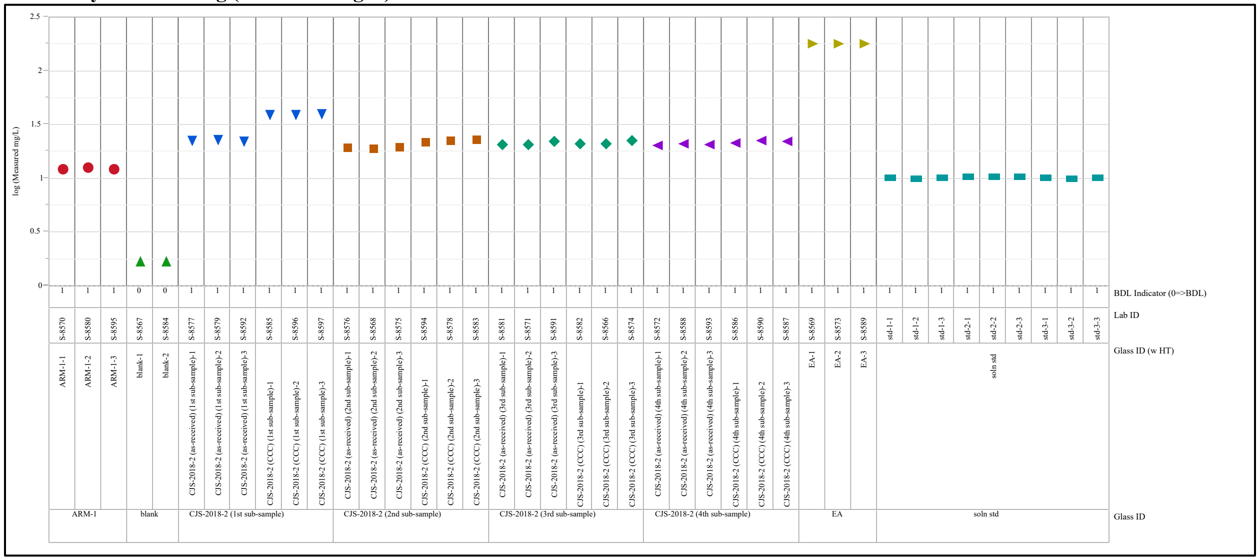


Exhibit C-2. PCT Measurements for Each Glass Subsample Grouped by Heat Treatment (continued)

Analyte=Na
Variability Chart for log (Measured mg/L)

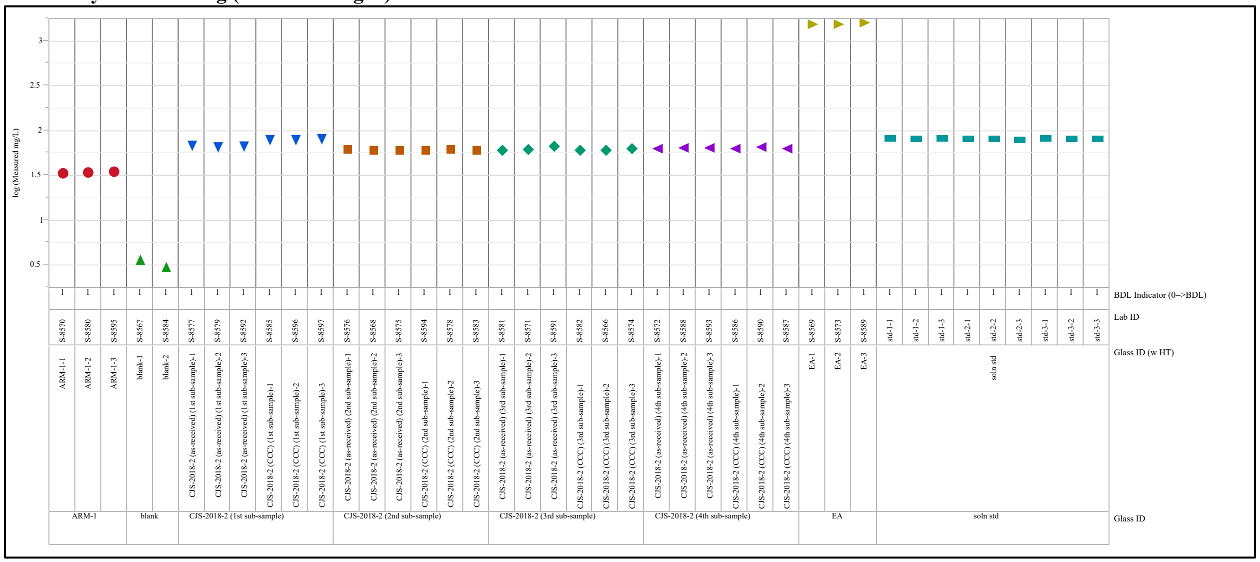


Exhibit C-2. PCT Measurements for Each Glass Subsample Grouped by Heat Treatment (continued)

Analyte=Si
Variability Chart for log (Measured mg/L)

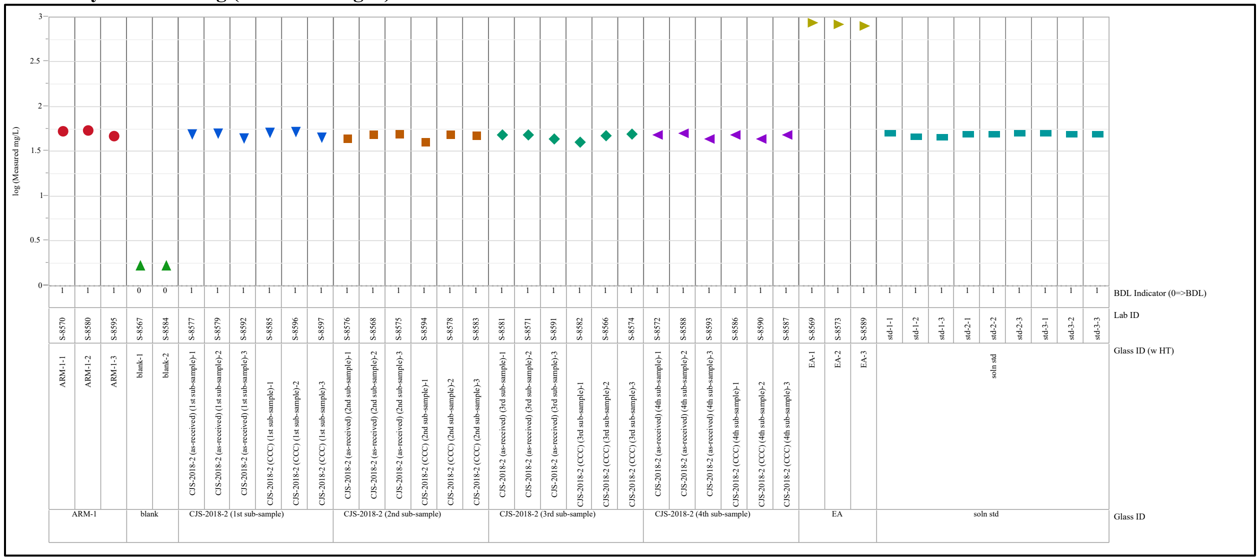
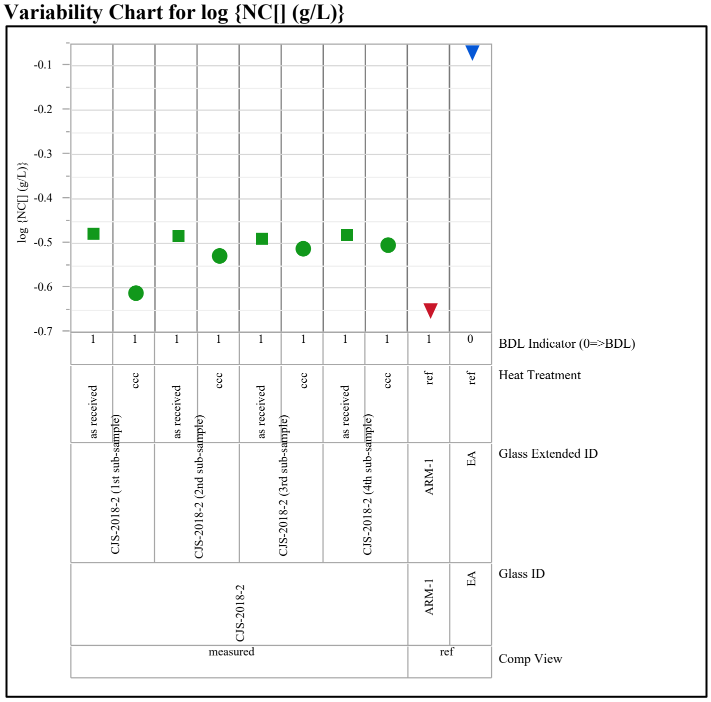


Exhibit C-3. Plots of Normalized PCT Results for Each Subsample Grouped by Heat Treatment

Analyte=Al
Variability Chart for log {NC[] (g/L)}



Analyte=B
Variability Chart for log {NC[] (g/L)}

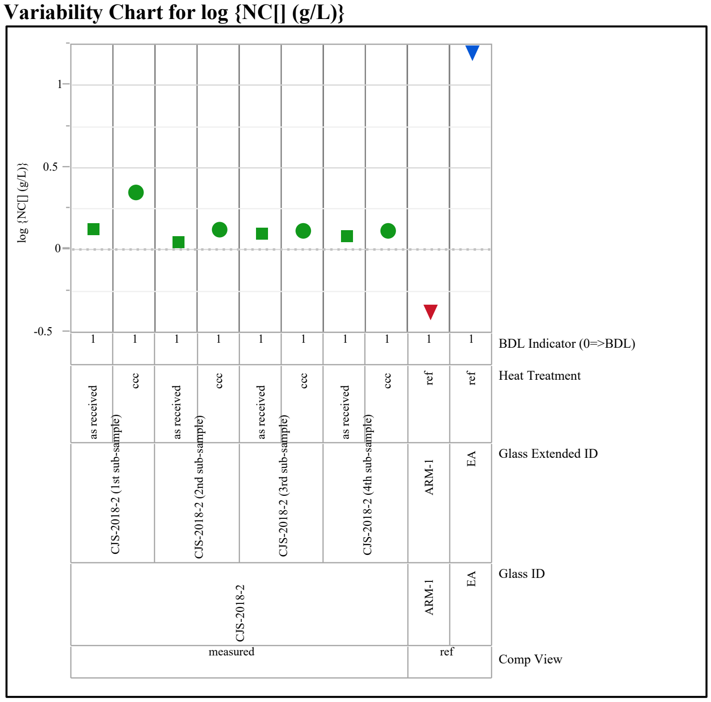
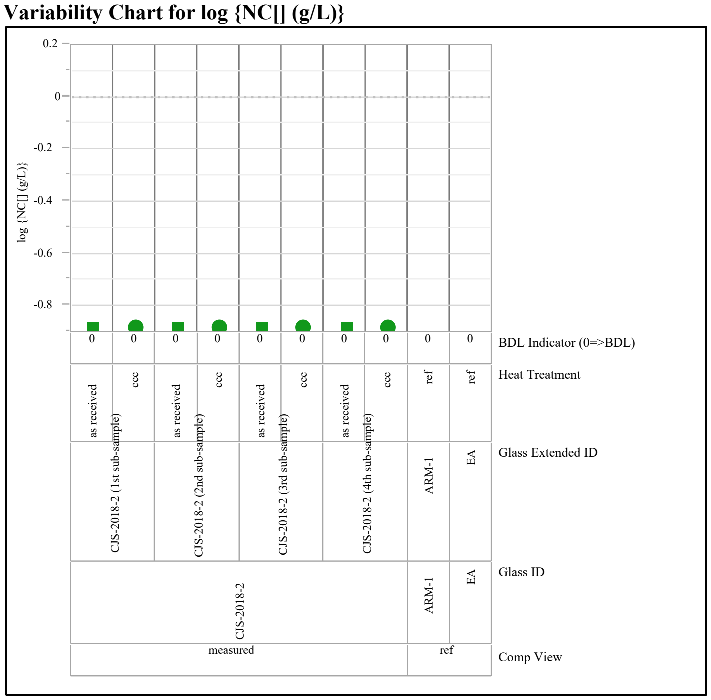


Exhibit C-3. Plots of Normalized PCT Results for Each Subsample Grouped by Heat Treatment (continued)

Analyte=Cr
Variability Chart for log {NC[] (g/L)}



Analyte=Fe
Variability Chart for log {NC[] (g/L)}

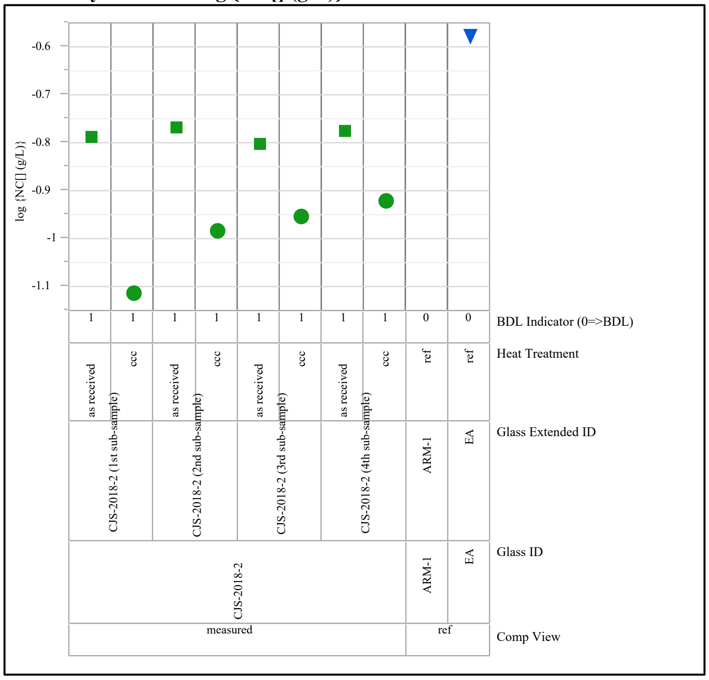
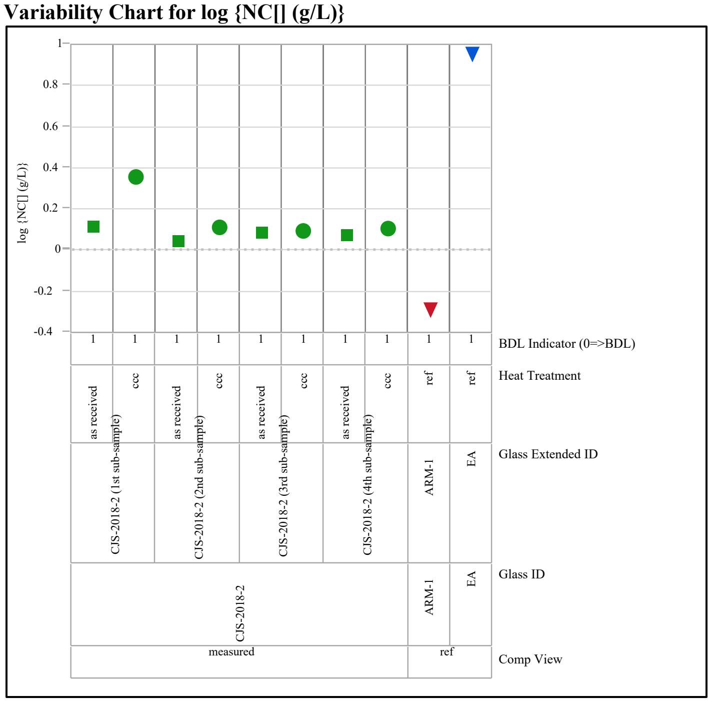


Exhibit C-3. Plots of Normalized PCT Results for Each Subsample Grouped by Heat Treatment (continued)

Analyte=Li
Variability Chart for log {NC[] (g/L)}



Analyte=Na
Variability Chart for log {NC[] (g/L)}

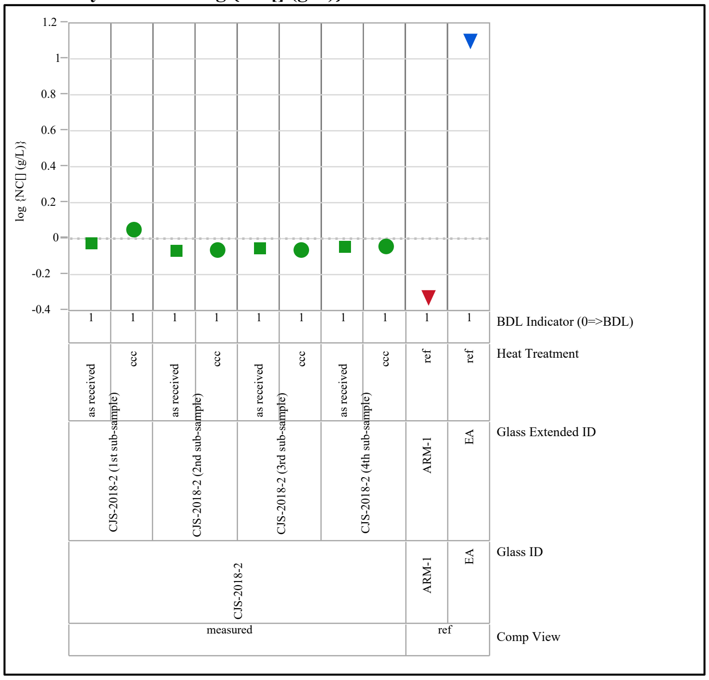
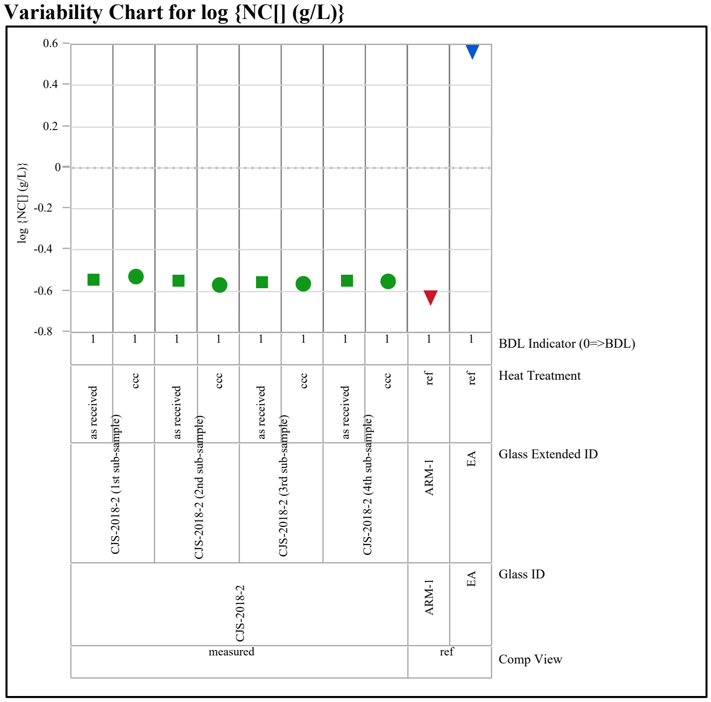


Exhibit C-3. Plots of Normalized PCT Results for Each Subsample Grouped by Heat Treatment (continued)

Analyte=Si
Variability Chart for log {NC[] (g/L)}



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